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VOLUME 26 OCTOBER 18, 1937 NUMBER 21

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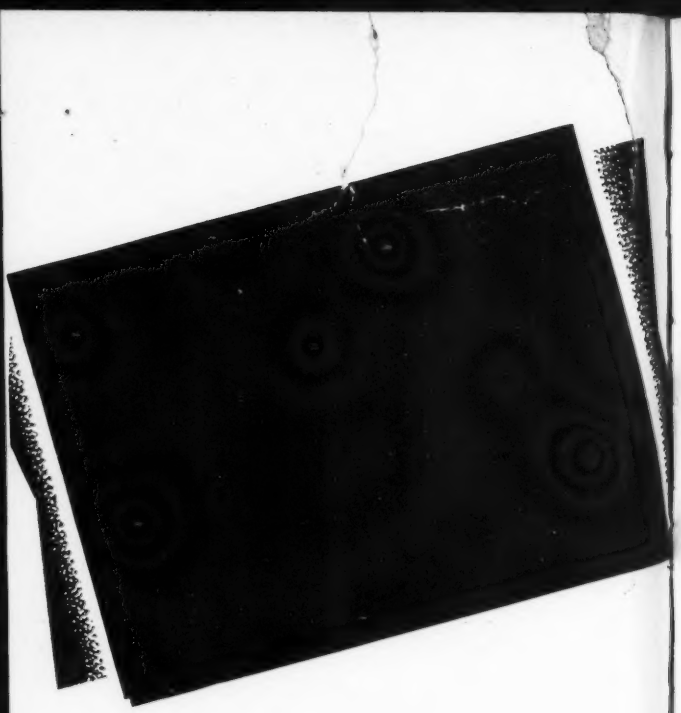

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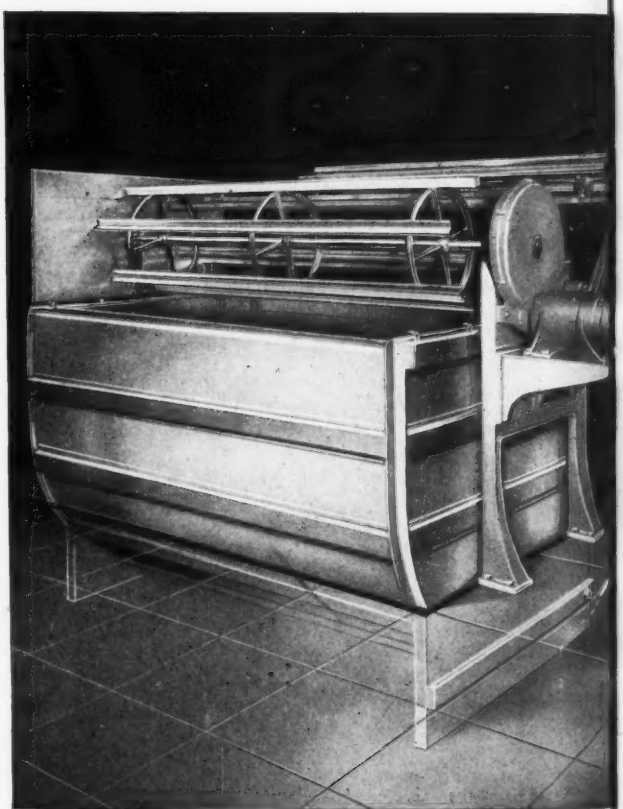

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Official Publication of the American Association of Textile Chemists and Colorists

THE OBJECTS OF THIS ASSOCIATION ARE:

"To promote increase of knowledge of the application of dyes and chemicals in the Textile Industry.

"To encourage in any practicable ways research work on chemical processes and materials of importance to the Textile Industry.

"To establish for the members channels by which the interchange of professional knowledge among them may be increased."

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AMERICAN
DYESTUFF REPORTER

VOLUME 26 OCTOBER 18, 1937 NUMBER 21

The Application of Logwood
to the Rayons

RAYMOND L. DREW*

LOGWOOD, the most important dyewood extract, has been used many years for the dyeing of silk and other natural fibers. It has been used in England as early as the 16th century and in this country, in some form or another, since the earliest times. A very interesting historical discussion may be found in an article by C. D. Mell, published in the *Textile Colorist*, No. 53, 1931. The material here is traced back to the time of its discovery and many interesting incidents in its career are given.

The discovery of synthetic dyestuffs and their later development and general use caused little if any loss in the amount of the various logwood extracts consumed. Their low price and their well known shade held these products in favor. With the advent of synthetic fabrics, however, logwood began to lose ground as a dyestuff for the textile field because of application difficulties and the lack of any practical and workable dyeing formula.

It is therefore natural to find that considerable technical work has been carried on in an endeavor to apply logwood and logwood products to the rayons. This work has been carried on by the manufacturers of logwood and also by those interested in its application, for the dyers have long considered the color of logwood very agreeable and desirable. The synthetic dyestuffs which have been applied to the rayons have been tolerated not because of any beauty of shade or ease in working properties but mainly for the reason that they could be applied commercially to these fibers which ordinarily have poor affinity for dyestuffs.

PATENTED METHODS

During the past ten or fifteen years there have been patented a number of processes which claim to make possible the production of good blacks by the use of logwood on cellulose acetate fabrics. The Bedford patent, U. S. No. 1,657,656, issued January 31st, 1928, cites a method which is general and which seems to give fair results on straight acetate rayon. On mixed fabrics, comprised of acetate and viscose, the results are apparently not so satisfactory.

This process generally consists in treating the fabric with a solution of the dyewood extract and then passing the impregnated cloth through a bath of sodium bichromate. Many agents may be added to the bath to form some improvement and a later patent, U. S. No. 2,042,802, to Herbert Platt, dated June 2nd, 1936, mentions the use of an alkali oxalate in this capacity. Other materials including acetic acid and borax have been used as assistants in the bichromate bath.

As opposed to this method, another system which depends upon mordanting the fabric with some type of metallic salt and then treating with logwood according to conventional dyeing practice has been patented and put to some commercial use. Many metals are suggested for the mordanting bath but iron seems by far the most practical.

In a French patent (1st Add., No. 39,164) to Henry Dreyfus, granted June 23, 1931, there is employed a process which makes use of a gelatinous paste of a metallic hydroxide formed by heating the alkaline precipitate in the presence of hydrochloric acid for some time at a temperature of 50° to 70° C. After the mordanting treatment by immersion in this bath, the material is dyed with haematein crystals to a deep black. The patent also mentions the use of other types of dyes such as Anthracene Blue WG and Gallocyanin. While this patent pertains especially to titanium and aluminum, the inventors claim that other metals may be used.

In this laboratory we have had very little success with this method in using logwood as we find considerable difficulty in producing a heavy black shade. This is probably because of the lack of affinity of the type of iron produced for the acetate fiber. Or it is possible that this type of iron, which is produced under these conditions, is not suitable as a bottom for logwood.

THE USE OF FERRIC CHLORIDE

In 1930 a patent was granted to Rivat, Cudgene and Dreyfus, U. S. No. 1,779,494, which covers the use of

* Research Dept., American Dyewood Co.

a mordant comprised of a strong solution of ferric chloride. The inventors recommend a strength of 40 to 55 per cent and a treatment of 40° to 60° C. for one hour. After washing, the material is dyed in a logwood or haematein solution as usual. The strong ferric chloride solution has considerable action upon metals of construction and it is also difficult to contain in wooden tanks. This, naturally, places considerable handicap upon the method and if the mordant solutions are reduced in strength to somewhat overcome this, they become very much slower in their action so that it is necessary to increase the time of fabric immersion to 12 or 15 hours. This, of course, presents a further disadvantage. The black, by this method, on mixed goods is satisfactory and we believe that the process has at least been carried out commercially in a test operation.

THE HYDROUS OXIDES

The latest work upon this subject deals with the treatment of the fabric in the mordanting bath with a suspension of hydrous oxides, U. S. Patent No. 2,085,795, dated July 6th, 1937. Under this process it is now possible to very readily mordant the fabric in 15 to 20 minutes so that it will take up quickly and easily enough logwood or haematein to produce a beautiful logwood black. The active element in the mordant is the yellow hydrous oxide. In the French patent to Henry Dreyfus, mentioned above, there is chiefly formed the red hydrous oxide which is by no means as potent as the yellow type. It is, no doubt, this same active red element that brings about mordanting in the system as covered by the patent to Rivat, Cudgene and Dreyfus, No. 1,779,494. It is formed in small quantities when a ferric chloride solution of high concentration is made.

Since the red hydrous oxide is not nearly as effective as the yellow or beta oxide, it is obvious that some system, which would form in an abundant quantity, the yellow or more effective beta hydrous oxide, would be ideal as a mordanting material for cellulose acetate fabrics and it was with this in mind that this laboratory undertook considerable research and experimental work. Before this, our early work on the application of logwood to acetate rayon was devoted entirely to variations of the reverse process such as is covered by the Bedford patent.

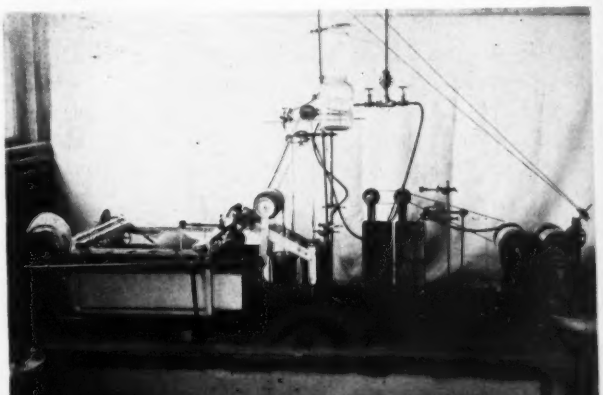
There are a number of methods given in the literature for the preparation of the beta iron oxide mono hydrates and in Meller, Treatise on Inorganic and Theoretical Chemistry, Vol. 13, as well as in "Constitution of Beta Iron Oxide Mono Hydrates," by I. N. Koethoff and B. Moskowitz, Am. Chem. Soc.—Vol. 58, No. 5, Page 777, some interesting information is given. All of the published work to date, however, deals with the preparation of the pure products by dialysis after formation in very dilute solution and the substances are not suitable for the practical operation of mordanting. The impure peptized colloidal suspension in rather concentrated form is required.

It was noted that when a rather strong solution of ferric chloride was boiled for a considerable period of time, its action as a mordant for acetate rayon was considerably increased. That is, acetate rayon steeped in material took up, in a given time, considerably more iron. After standing, however, this solution lost its potency because of the reversibility of the reaction and the loss of the active ingredient.

It was found that by treating a solution of iron chloride or nitrate preferably, although other salts can be used, with sufficient alkali in the form of the hydroxide to partially neutralize the iron and the material boiled for a considerable period of time so as to peptize the suspended hydrous oxide, that an excellent mordant for practical work was produced. As far as the active properties of the mordant are concerned, it is not important that the excess of liberated mineral acid be taken up with sodium acetate although for the formation of a less corrosive mixture, this extra reaction is helpful. In a mordant of this kind which would contain approximately 25 per cent of available iron salt, it is possible to get an iron deposit, expressed as Fe_2O_3, of approximately one-half per cent on the weight of the fabric in 15 to 20 minutes' immersion at 50° C. Rayon fabrics, especially the acetate, so impregnated may then be dyed in the usual logwood bath of 1 to 3 per cent and the usual rich black logwood shade obtained. Other dyewood extracts such as fustic and osage orange can be used for toning or altering the shade of the black. In addition to this it is also possible to tone the shade, if desired, by the addition of some aniline colors to the logwood bath.

SMALL SCALE OPERATION

In our laboratory work we have used a small machine such as that illustrated so as to obtain a continuous dyeing



From Left to Right: Box for continuous mordanting; counter current washing system; dye box; wash box; dryer

operation. It is designed especially for use with mixed goods for, in the case of straight acetate materials, the ordinary dyehouse jigger which has been equipped to handle this mordant may be used in the mordanting operation and after washing, the dyeing can be accomplished flat in another jigger. The problem, in the case of the

flat acetate fabric is not a serious one. On mixed goods this process is particularly satisfactory because it dyes both fibers to approximately the same shade and strength. This, in fact, is considered an important feature of the process.

The mordant bath, in our laboratory machine, is built according to the scheme used in the Van Vlaanderen weighting unit so that it is possible to get a fair speed of cloth through the machine and still obtain the 15 to 20 minute immersion required for satisfactory mordanting.

After the mordanting operation, a continuous countercurrent washing system is set up so as to remove, in a concentrated form, as much of the mordant as possible. This diluted mordant can be strengthened by evaporation and returned to the original mordanting bath so that only an extremely small quantity of the iron salts is lost. The greatest cost in mordanting is caused by the mordant which is mechanically lost and not by that which is held chemically in the fiber. It is therefore necessary, in a process of this kind, to take advantage of continuous operation so that mordant and dyestuff losses can be worked down to a minimum.

After the mordanting and washing process, the dyeing operation may be carried out in the piece or by a continuous system. This operation follows the conventional dyehouse procedure for logwood using, for heavy blacks, a 3 per cent standing bath of logwood or haematein depending upon the particular condition at hand.

An important feature of this process is its ability to dye the various fibers of mixed goods to about the same degree of shade and strength. The finished material possesses the usual characteristics of logwood dyed material as concerns its physical characteristics and tone of color. It should make possible, after its full development has been accomplished, a considerable saving in the cost of dyed fabrics which come within its range.

Physical and Chemical Textile Testing - IV

JOHN H. SKINKLE*

PART I—PHYSICAL TESTING

(Continued from October 4, 1937 issue)

CHAPTER 5—FABRIC TESTING

Introduction

BEFORE taking up the various tests usually given fabrics, it will be necessary to discuss briefly the various types of fabrics from the point of view of methods of manufacture, production of pattern, general properties of the finished product, and some of the terms used in referring to the fabric. These will be discussed only in a very elementary way because we are concerned primarily with the testing and not with production of the fabrics. Practical experience and the study of other texts are necessary to supplement this very elementary survey.

The textile fabrics may be classified as follows:

1. Felts—fabrics made by uniting loose fibers into a uniform sheet.
2. Knit goods—fabrics made by looping one yarn about itself.
3. Braids and laces—fabrics made by twisting many yarns about each other.
4. Woven goods—fabrics made by passing one set of yarns above and below another set of yarns at right angles.
   a. Tapes and ribbons—made very narrow and on special looms.
   b. Mechanical fabrics—ordinary wide goods of fairly simple weave where appearance is secondary to strength or other physical properties.
   c. Dress goods, suitings, coatings, poplins, broadcloth, etc.—of fairly simple weave but where appearance is more important.
   d. Print goods—of fairly simple weave but with a complicated color pattern applied by a printing process.
   e. Tapestries and other similar fabrics—of very complicated weave produced by complex looms or Jacquard looms. Appearance is usually much the most important quality in these goods.
   f. Pile fabrics such as plushes, velvets, corduroys, and carpets—fabrics in which some of the yarns are cut during or after weaving so that the cut ends form the face of the fabric.

Felts have no regular structure, consisting of masses of fiber (usually hair fibers) united together by chemical treatments. Tests made on felt are usually for content and are chemical and optical tests; thickness is the most important physical test with porosity sometimes determined.

Knit goods consist of a series of loops of a single yarn; other yarns may be used to make stripes but usually only one strand at a time. The loops extending lengthwise of the fabric are known as wales, and those crosswise of the fabric as courses. Knit goods are usually made in tubular

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form so that there are no edges. Hosiery and underwear form the bulk of the knit goods; so that, in general, appearance is the most important property. Physical tests, then, are not as important on knit goods as they come on the market; most of the testing of knit goods is done in the process of manufacture and are carried out on the yarn which goes into the goods.

Braids and laces, also, are used principally for ornamentation and again the most important tests are those made upon the constituent yarns. This class of fabric is, in point of quantity, relatively unimportant.

The woven goods are much the most important group of fabrics. They are made in a loom by arranging a number of yarns (the warp) under tension and capable of being lifted and dropped by the harness mechanism, each individual yarn of the warp (ends) passing between a set of combs called the reed. With some of the ends lifted and some dropped to form an angle (shed), a shuttle flies across carrying one yarn (pick); these cross yarns are known as the filling. Different patterns are made by putting in different yarns in the warp and in the filling, also by lifting the warp yarns in various combinations. The filling yarns change direction at each edge of the fabric and to keep them firmly in position at the edges, a special lot of warp yarns very close together (the selvedge) is put in; obviously the cloth is firmer and in general different in properties near the selvedge, so it is a general rule that no physical tests shall be made nearer than one-tenth the width of the fabric to the selvedge. The use to which the fabric is to be put governs the tests which are given it; in any case an analysis of the constituent yarns and their arrangement in the fabric should be made, preferably by an experienced designer; the tests which we are to discuss assume that the design of the cloth has already been passed upon and found correct.

Mechanical fabrics are fabrics to be used where appearance is unimportant. Usually strength is of the most importance, but sometimes other properties are equally or more desirable. In aeroplane wing fabric or balloon cloth, the permeability is an important factor, which in turn is dependent upon the size and closeness of the yarns in the cloth. In bandage cloth or toweling, the ability to absorb liquids is very important; in canvas and umbrella cloth, waterproofness is most desirable.

In cloth to be used for clothing, both strength and wearing properties are important but must be combined with a good appearance, good draping qualities, a pleasant "handle", and preferably a light weight; other properties which may be desirable in special clothing are waterproofness, coolness or warmth, or ability to resist creasing.

In pile fabrics, in addition to appearance and construction, the most important physical property of the cloth is the ability of the cut ends or pile to remain upright under a load, or if bent by the load to regain its upright position when the load is removed. This involves the study of the thickness of the fabric under varying loads, also the resiliency of the fabric.

To study every physical property of every known fabric would be out of the question. We will therefore take up only the more important properties which should, however, suggest a method of approach to the determination of other properties.

YARN COUNT

This determination measures the number of warp yarns per linear inch (ends per inch or sley) and the number of filling yarns per linear inch (picks per inch or shot). In the case of knit goods, the corresponding values are wales per inch and courses per inch.

There are four general methods of determining yarn count:

1. Unraveling the cloth.
2. Use of the pick glass.
3. Mechanical pick counter.
4. Fabric projector.

Unraveling the cloth is used principally on dense fabrics with a felted surface where the separate yarns are hard to distinguish; it is also sometimes done where, in addition to yarn count, it is also desired to know the relative weights of warp and filling. The pick glass is a small magnifying glass in a stand over a square exactly one inch each way (or sometimes one-half inch each way), so that if all the yarns in the field are counted the number of yarns per inch is directly determined; this is the usual method used. The mechanical pick counter, such as the Suter, consists of a small microscope arranged to travel horizontally on a calibrated track; in the field of view is a needle point; it is placed on the cloth with the pointer at zero and traversed across, counting the yarns as they pass under the needle point, for a known distance, and the number of yarns per inch calculated. The projector is simply a slide projector in which the cloth is placed between two plates with an opening of known dimensions, the number of yarns may then be easily counted on the screen; it is useful principally to avoid eyestrain.

If the pick glass is used, it is convenient to use a light table, a box with a ground glass top and containing several electric bulbs. The A.S.T.M. specifies that the number of ends per inch and picks per inch shall be counted in five different places, not counting the same threads twice, and of course not near the selvedge. If there are fewer than 25 threads per inch, the number of threads in three inches shall be counted each time and the result divided by three. If the fabric is less than three inches wide, the total number of ends shall be counted and divided by the width.

If the pattern of the cloth is very complicated, the number of yarns per inch has little meaning and the number of yarns to a pattern should be determined, this is really a job for a designer.

REFERENCES:

[1]Haven, G. B. Mechanical Fabrics. John Wiley and Sons, Inc., New York (1932).

[2]A.S.T.M. Standards on Textile Materials. A.S.T.M., Philadelphia (1936).

WEIGHT

Two different weights are in use and may be determined in evaluating cloth: weight per unit area (ounces per square yard) and weight per unit length (ounces per running yard). Obviously if either one and also the width of the cloth is known, the other may be calculated (assuming the selvedge to have a negligible effect) by the formula:

$$S = 36R/B$$

and $$R = SB/36$$

where S = weight per unit area (ounces per square yard)

R = weight per unit length (ounces per running yard)

B = width of cloth in inches

Both weight determinations are based on cutting out pieces of cloth of known area and weighing. The value of S may then be calculated from the formulae:

$$S = \frac{36\text{x}36}{A} \times \frac{G}{437.5} = 2.962G/A$$

or

$$S = \frac{36\text{x}36}{A} \times \frac{G'}{28.35} = 45.71G'/A$$

where A = area of sample in square inches

G = weight of sample at standard regain in grains

G′ = weight of sample at standard regain in grams.

There are three general methods in use for determining the weight:

1. Weighing a large number of small areas; e.g., five pieces each two inches square.

2. Weighing one fairly large area; e.g., a piece 5″ x 4″.

3. Weighing a piece the full width of the cloth and one yard long. This gives the value of R directly.

In either of the above methods enough sample must be taken to give at least twenty square inches. The weighing should always be done when oven-dry and the results calculated to standard regain.

The cutting-out of the samples may be done by marking off the area with a ruler and cutting out with shears; but if many determinations are to be made, it is better to use steel or glass templates, cutting around the templates with a sharp razor blade. Where a large number of small samples are to be cut out, steel dies may be used.

Many cloths are bought and sold by a description which includes the weight per running yard, this is possible only when a standard width is agreed upon. Woolen and worsted cloths are based upon a 54 inch width, so that a 16-ounce worsted fabric is one of which a piece 54 inches wide and one yard long would weigh 16 ounces at standard regain. Similarly, cotton duck is based on a width of 22 inches, and a 16-ounce duck would be one of which a piece 22 inches wide and one yard long would weigh 16 ounces at standard regain. Ducks are also given a number which is equal to 19—R so that the above duck is 19—16 = No. 3 duck, a 19-ounce duck is No. 0 or 1/0, a 20-ounce duck is No. 00 or 2/0, a 21-ounce duck is 3/0, and so on.

The following examples will make clear the use of the formulae:

A piece of cotton cloth 4″ x 5″ weighs 1.55 grams when oven-dry. What is the weight per unit area?

G′ = 1.55x1.065 = 1.65 grams

A = 4x5 = 20 square inches

S = 45.71x1.65/20 = 3.77 oz./sq. yd.

A sample of woolen cloth is to be tested for weight. Five pieces each 2″x2″ are cut out and weighed together; when over-dry they weigh 112 grains.

A = 5x2x2 = 20 square inches

G = 112x1.13 = 126.7 grains.

S = 2.962x126.7/20 = 18.7 oz./sq. yd.

R = 18.7x54/36 = 28.1 ounces per running yard of cloth 54 inches wide.

A sample of duck is 21 inches wide and a length of 12 inches weighs, when oven-dry, 144 grams. What is the number of the duck?

A = 21x12 = 252 square inches

G′ = 144x1.065 = 153.3 grams.

S = 45.71x153.3/252 = 27.8 oz./sq. yd.

R = 27.8x22/36 = 17 oz./running yd.

19 — 17 = No. 2 duck

REFERENCES:

[1]A.S.T.M. Standards on Textile Materials. A.S.T.M., Philadelphia (1936).

CRIMP

Crimp may be defined as the deviation from straightness of yarns as they lie in the cloth, expressed as percentage of length in the cloth. Take-up is the deviation from straightness of the yarns as they lie in the cloth, expressed as percentage of the original length of the yarn before weaving. The interrelationships of crimp and take-up may be shown by the following formulae:

Let L = length of yarn before weaving

L′ = length of yarn in the cloth

C = crimp in %

T = take-up in %

Then $$C = \frac{L - L'}{L'} \times 100$$

$$T = \frac{L - L'}{L} \times 100$$

$$T = \frac{100C}{C + 100}$$

$$C = \frac{100T}{100 - T}$$

Since the process is sometimes reversed and it is desired to know the original length of the yarn, from the above formulae we obtain:

$$L = \frac{L'(C + 100)}{100}$$

$$L = \frac{100L'}{100 - T}$$

In a fabric just off the loom, the warp threads generally have the most crimp because the filling yarns are drawn straight through the shed and the warp yarns are folded over and under the filling yarns by the up-and-down motion of the harnesses. Finishing operations such as stretching, calendering, and tentering may change the crimp. Crepes are different as regards crimp; in these fabrics the filling yarns are excessively twisted and tend to kink, the warp yarns take up some of this kink and rest in the cloth in a fairly straight condition.

A fabric which is to have all or most of the stress applied in one direction (such as duck belting) should have the minimum crimp in that direction; but fabrics (such as balloon cloth) which are subject to equal stress in all directions should have the same crimp in both warp and filling. This last fabric is known as a "square" fabric; in general the difference between warp and filling crimp is known as "off-square"; for example, if the warp crimp is 6 per cent and the filling crimp is 4 per cent, the cloth is 2 per cent off-square.

It will be noted that crimp and take-up are the same quantity referred to different bases. The crimp is based on the length in the cloth and is of interest to the tester and the finisher; the take-up is based on the length before weaving and is of interest to the weaver and yarn manufacturer.

The principle of the crimp determination is very simple: with a fine pen and a rule, lines are drawn on a piece of cloth L′ distance apart, some of the yarns are raveled out, the yarns are straightened without stretching, and the distance between the ink marks (L) is noted; the crimp is then calculated from the formula. The difficulty lies in the straightening of the yarn without stretching it; to do this three methods are available:

1. Straighten by hand—this is inaccurate since we do not know the force applied.

2. Straighten by a standard weight—this is satisfactory if we know what weight to use, the determination of this weight will be given later.

3. Determine the straight length from the load-elongation curve—this is the most accurate method and the one which we shall take up in detail.

The usual method of crimp determination by load-elongation data is as follows: Make parallel ink lines on a piece of cloth L′ distance apart, unravel five yarns and test one at a time. Clamp the yarn at one of the ink spots, allowing the yarn to hang vertically in front of a scale; hang a small clamp of known weight on the yarn at the other ink spot, read the elongation at this load. Apply successively small loads, reading the elongation at each new load until it is obvious that each additional load gives the same additional elongation. Plot the elongation horizontally and the load vertically to obtain a curve similar to Figure 24. In this curve, there is a curved portion (OB) and a straight line portion (BC). The region OB represents the removal of the crimp, the region BC represents the stretch of the yarn; if there

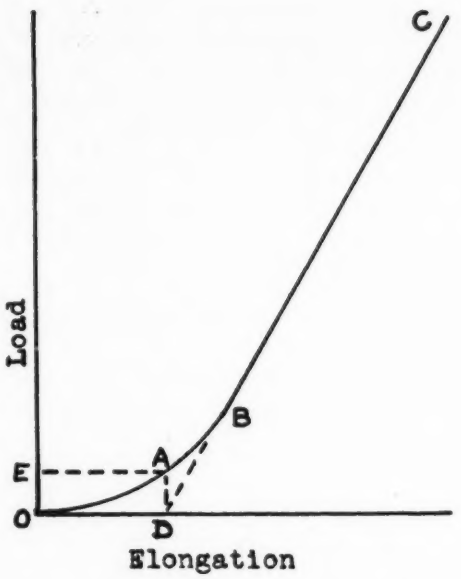

Fig. 24—Load-Elongation Curve for Crimp Determination

were no crimp, the curve would be all the straight line DC, therefore the distance OD represents the elongation of the yarn due to the removal of the crimp; then the original length L′ plus the value OD is the length of the yarn before weaving (L) or

$$L = L' + OD$$

This method was originally devised by the Bureau of Standards who loaded the yarn by attaching a light weight scale pan and adding weights; this method has been improved by using the chainomatic principle, loading the yarn by lowering a chain so that an increasing number of links of the chain are held by the yarn (Figure 25). A simple and inexpensive apparatus may be made as follows: On a vertical board, fasten graph paper marked off in inches and tenths, fasten a small clamp at the top to hold the yarn. Obtain a small paper clamp with jaws ¼″ wide and with a hook attached, such as are sold for use in schools to hang papers on a piece of cloth; obtain also a number of paper clips. Weigh the small clamp, and a number of the paper clips. Hang the yarn in the clamps, read the extension; add the paper clips, one at a time, reading the extension. A typical determination is given:

Weight of small clamp = 0.35 grams
Weight of 5 paper clips = 3.90 grams
Weight per paper clip = 0.78 grams

Weights added	*Elongation —inches*	*Load —grams*
0	0	0
clamp	.21	.35
clamp + 1 clip	.37	1.13
clamp + 2 clips	.41	1.91
clamp + 3 clips	.45	2.69
clamp + 4 clips	.49	3.47
clamp + 5 clips	.53	4.25

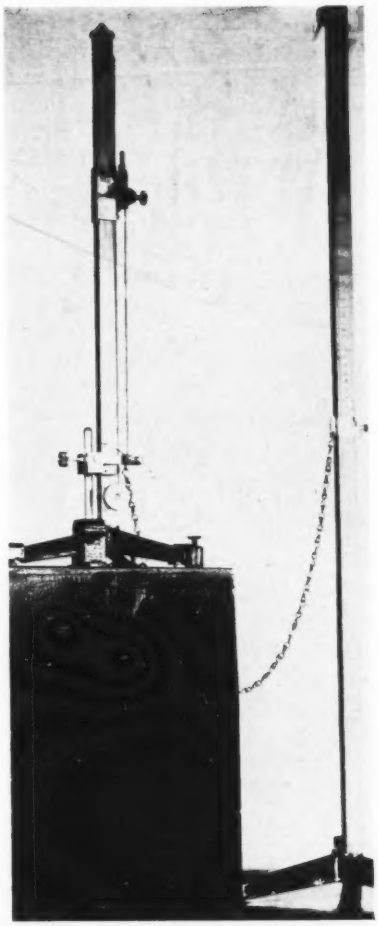

Fig. 25—Crimp Determination Apparatus (Chainomatic Type)

On plotting the the above and extending the straight line portion, it was found that OD = 0.31 inches. Therefore

$$L' = 10 \text{ inches} \qquad L = 10.31 \text{ inches}$$
$$C = \frac{0.31 \times 100}{10} = 3.1\% \text{ crimp}$$

If the crimp is to be determined by the length with a standard weight applied, it is necessary to have one weight for each type of yarn and fabric; this is satisfactory in a mill where only one class or a few classes of goods are being made. The standard weight to be used is determined from the load-elongation curve by projecting from D to the curve and from the curve over to the load axis, in Figure 24 the distance OE represents the weight which would be just sufficient to remove the crimp. Another way of determining the standard weight is to use the A.S.T.M. formula:

$$\text{If } L' = 8 \text{ inches, then}$$
$$OE = \frac{131}{\text{Typp number}}$$

This formula is merely an approximation and is not as accurate or desirable as the previous method.

It has been found that temperature and relative humidity have only a very slight effect on the crimp, so that standard conditions are not necessary in crimp testing.

Crimp may also be determined from the strength-stretch curve which will be taken up later, but the results are not as accurate as the methods just described due to the small scale of the curve.

A typical problem involving crimp follows: A sample of cotton cloth 6″ square is picked apart and there is obtained 360 warp yarns which weigh 0.955 grams when oven-dry and 320 filling yarns which weigh 1.150 grams when oven-dry. A crimp determination gave 4.2 per cent crimp in the warp and 3.6 per cent crimp in the filling. What is the yarn count, the weight of the cloth, and the counts of the warp and filling?

Yarn count:

Warp 360/6 = 60 ends per inch
Filling 320/6 = 53.3 picks per inch

Weight:

Warp = 0.955 grams
Filling = 1.150 grams
Total 2.105 grams

2.105 x 15.43 x 1.065 = 34.6 grain at standard conditions

S = 2.962G/A = 2.962 x 34.6/36 = 2.84 ounces per square yard

Counts:

Warp

$L' = 360\text{x}6 = 2160 \text{ inches} = 60 \text{ yards}$

$L = \frac{L'(100+C)}{100} = \frac{60\text{x}104.2}{100} = 62.6 \text{ yards}$

$W = \frac{0.955\text{x}15.43\text{x}1.07}{1} = 15.8 \text{ grains}$

$N = \frac{L \times 7000}{W \times H} = \frac{62.6 \times 7000}{15.8 \times 840} = 33.0\text{'s}$

Filling

$L' = 320\text{x}6 = 120 \text{ inches} = 53.3 \text{ yards}$

$L = \frac{53.3\text{x}103.6}{100} = 55.2 \text{ yards}$

$W = \frac{1.150\text{x}15.43\text{x}1.07}{1} = 19.0 \text{ grains}$

$N = \frac{55.2 \times 7000}{19.0 \times 840} = 24.2\text{'s}$

REFERENCES:

[1]A.S.T.M. Standards on Textile Materials. A.S.T.M., Philadelphia (1936).

[2]Haven, G. B. Mechanical Fabrics. John Wiley and Sons, Inc., New York (1932).

THICKNESS

The determination of the thickness of a textile material is a delicate operation because of the ease of compression of most fabrics; for this reason, it is seldom done except upon felts, ducks, or other fairly rigid fabrics. If the thickness of a soft material must be determined, it is bet-

Fig. 26—Thickness Gauge

ter to do it microscopically on a cross-section of the fabric. This is especially true of pile fabrics.

The usual type of thickness gauge (Figure 26) has a broad anvil upon which a presser foot is pressed by a spring. The sample is placed on the anvil and the presser foot is lowered onto the sample by releasing the raising lever very slowly, the dial then indicates the number of thousandths of an inch between the anvil and the presser foot.

Haven has listed the variables which affect the result and has attempted to standardize these variables in the following manner:

1. Size of presser foot—greater the size, the less it will sink into the fabric; one inch suggested.

2. Form of presser foot—Haven suggests circular for cloth and square for cords.

3. Weight on presser foot—greater the weight, the more it will sink into the cloth; two ounces suggested.

4. Velocity of presser foot—faster the foot is allowed to drop, the more it will penetrate the fabric. Haven suggests a clock-work mechanism which would lower the presser foot at the rate of twelve inches per minute.

5. Time elapsing during measurement—presser foot does not come to equilibrium immediately; five seconds suggested.

6. Stability of the machine—jarring will cause the foot to penetrate deeper; a solid support is necessary.

7. Condition of the specimen during measurement—fabric should be free of wrinkles but not under tension.

The A.S.T.M. has standardized on the following method of using the thickness gauge: The presser foot shall be circular and 0.375 ± .001 inches; the presser foot and anvil shall be plane and parallel to within .0001 inches; the total weight on the presser foot shall be 6.0 ± 0.1 ounces (3.4 pounds per square inch). The fabric shall be placed on the anvil smooth and without tension; the presser foot shall be lowered gradually (without impact) and allowed to rest ten seconds before the dial is read. Not less than five measurements shall be made on a sample and the results averaged.

The A.S.T.M. also modifies the above specifications in the case of pile fabrics and wool felts as follows:

Total thickness of pile floor coverings
- Pressure = 0.100 ± .001 pounds per square inch
- Diameter of foot = 3.00 ± .01 inches

Thickness of backing of pile floor coverings
- Pressure = 0.750 ± .001 pounds per square inch
- Diameter of foot = 1.00 ± .01 inches

Thickness of wool felt
- Pressure = 1.0 pounds per square inch
- Diameter of foot = 1.129 inches
- Time before reading = 30 seconds.

In spite of all the above precautions, however, the values obtained are of questionable accuracy, although they do have some value in the comparison of different samples done on the same apparatus, by the same person, and using the same technique.

REFERENCES:

[1]A.S.T.M. Standards on Textile Materials, A.S.T.M., Philadelphia, 1936.

[2]Haven, G. B., Mechanical Fabrics, John Wiley and Sons, Inc., New York, 1932.

(To be continued)

CORRECTION

Skinkle—"Physical and Chemical Textile Testing"

American Dyestuff Reporter; October 4, 1937; page 608; lower right-hand column should read as follows:

$$W = \frac{1 \times 18.0 \times 15.4 \times 1.07}{6} = 49.4 \text{ grains}$$

Length of ply yarn before cording

$$= \frac{120}{1 - 6/100} = \frac{120}{.94} = 127.7 \text{ yards}$$

Length of single yarn before plying

$$= \frac{127.7}{1 - 2/100} = \frac{127.7}{.98} = 130.2 \text{ yards}$$

$$\text{Then } N = \frac{L}{W} \times \frac{7000}{H} = \frac{130.2}{49.4} \times \frac{7000}{840} = 22.0$$

The cord is 22/2/3 cotton.

Historical Review of

Aniline Black

AMELIA E. KARR

ANILINE black is the embodiment of the genius and labor of numerous chemists. Several early investigators succeeded in obtaining the chemical compound which we now designate as aniline. Since, however, the method and the source used differed with each individual chemist, each thought that he had discovered a new and unique chemical compound and named it accordingly. Unverdorben in eighteen twenty-six isolated an oily substance; a new chemical compound, thought he, and named it crystalline. Then Runge in eighteen thirty-four isolated an oily substance from coal tar oil and he, too, was of the opinion that here was a distinctly new chemical compound and consequently named it kyanol. Kyanol had a most interesting chemical property. On the addition of chromic acid it formed a green precipitate. This colored precipitate destined several years later to play a leading role in the formation of aniline black was recorded by Runge as one of the products of the reaction. Merely that.

Aniline was obtained from indigo by Fritsche. He called the oil, aniline. To Fritsche this oily chemical compound aniline was fascinating, and he proceeded to probe more deeply into its nature. In eighteen forty when a solution of an aniline salt was treated with chromic acid a dark green precipitate formed. Repetition yielded a blue black solid. This was considered an adequate test for aniline. These two different colored substances held Fritsche's attention. Three years later he experimented with them again. This time their chemical constitution was the object of his research. He made little headway in this direction but did find however, that the amount of chromic acid added determined the color of the final aniline compound formed.

That same year Fritsche showed that potassium chlorate and hydrochloric acid mixed with alcohol and aniline salt resulted in the formation of the colored precipitate that was destined many years later to become what is now known as aniline black.

To professor Eilhard Mitscherlich, famed crystallographic and mineralogic chemist, belongs the honor of having prepared the chemical compound nitro-benzene from benzene that he had in turn obtained from benzoic acid. Nitro-benzene was important, at the time, in perfume chemistry. It possessed an odor similar to that of bitter almonds. That it could ever play any part among dyestuffs was not very clearly seen even when Zinin showed that aniline could be produced from nitrobenzene by a chemical reaction. Aniline had not at that time been revealed as a progenitor of synthetic dyestuffs.

In eighteen forty-three Hoffmann turned his attention to this dyestuff progenitor and it was his researches that brought to light the singular fact that the chemical compound that had been given so many different names by research workers was one and the same compound, aniline. This oily substance was at that time a mere laboratory rarity. Interesting only as a part of pure science. Until it could be made available in commercial quantities not much could be hoped from it and even if it were thus available no one had any idea what to do with so much of it. Despite this, research continued. In the year eighteen fifty-four, Bechamp showed that aniline could be made on a commercial scale.

With comparatively large quantities of aniline available, industry was ready for something big to develop. It did. Only two years later it happened. The occurrence was Perkins' discovery of the first commercially successful aniline dyestuff, mauve. It was the tremendous success of this dyestuff that inspired so much of the research activity that took place in the dyestuff field thereafter.

Five years later four chemists, Crace, Calvert, Clift and Lowe experimented with aniline. They repeated Fritsche's experiment, the one in which potassium chlorate reacted with aniline hydrochloride. They, too, obtained a dark green or blue black precipitate depending on the conditions of the experiment. They went one step further and tried to produce the green which they called emeraldine on the fiber itself. They obtained a patent for producing on textile materials an insoluble green color, emeraldine, which could be transformed by alkalis into blue.

A year or two after, in January, eighteen sixty-three, Lightfoot of Accrington obtained a patent for the production of a dark green that might be employed as a black. The firm of J. J. Muller & Co. of Basle bought it. In this patent it was stated that copper chloride, hydrochlorate of aniline, acetic acid to liberate the chloric acid and starch paste must be used. The color was claimed to be developed in oxidizing chambers by the reaction of the potassium chlorate and the chloride of copper. This was employed for printing and was blacker than the emeraldine of Crace et al.

On April twenty-second, eighteen sixty-three, Cordillot, experimented with the greatest of secrecy. He did not even enter a statement about it in his laboratory notebook. Aniline salt, sal ammoniac, ammonium chloride, potassium chlorate and ferric chloride were made into a mixture for printing. First results were unsatisfactory but more experiments followed and on January sixteenth, eighteen sixty-four, a new aniline black was discovered. It became

known as the ferricyanide or prussiate black. This was used on a large scale and was most successful.

Before the world became aware of Cordillot's work Lauth had come forward with his method for the making of aniline black. He proposed the use of copper sulfide. This had never been used before. The copper sulfide used in this aniline black printing composition does not act as an oxidizing agent but merely as a catalyst. Soluble copper salts are too active and it is because of the insolubility of the copper sulfide and its slow oxidation to copper sulfate that a slow formation of the color is obtained that will not tender the fiber. This non-weakening of the fiber is one of the advantages of this method.

To get a black of still greater intensity no oxidizing agent such as a solution of potassium dichromate must be used. However, if too much of this chemical compound is used, a red tint will be observed and instead of a jet black a reddish black results. Acids will change the reddish dyestuff into a deep black. If excess acid is used the emeraldine appears. The deep black can be formed from this by the employment of alkalis of any kind.

Considering the tendering of the fiber Camille Koechlin suggested that the tartrate of aniline be used in place of the hydrochloride. This does not attack the fiber. However, to get the desired results it is necessary to add ammonium chloride.

Until this time aniline black had been made in the powdered form and sold to be used in printing compositions, although Lightfoot is said to have had the foresight to know that it could in some way, through modifications, be applied to dyeing proper. It was the Frenchman, Bobeuf, who first patented a method for dyeing. In eighteen sixty-five Bobeuf's patent stated that aniline black could be used in dyeing silk, wool, or other animal fibers, cotton, hemp, yarn, linen or other ligneous or vegetable substance.

His method was to use two baths. It made no difference which one was utilized first. Either the dichromate or the hydrochloride of aniline. This method was patented July fifteenth, eighteen sixty-five. During that same year on August the fifth Alland received his patent for the use of aniline black in both the dyeing and printing of textiles. Bobeuf included other chemical compounds besides potassium dichromate. His experiments dealt with the oxalates, arsenates and others and showed that each gave an aniline black.

The idea had developed that a metal, particularly copper, was an essential in the preparation of aniline black. It was shown however that aniline black could be produced by the use of hydrosilico-fluoric acid. A few drops of hydrochloric acid must be used to cause precipitation. Another proof of this was brought forth years later in eighteen seventy-five through experiments done by Coquillon. These experiments in which aniline sulfate in concentrated solution was subjected to the action of the electric current using platinum electrodes, showed a black mass around the positive pole after the current had passed through the solution from twelve to twenty-four hours. To be certain that the aniline black was formed without any influence whatever from metals Coquillon substituted carbon electrodes for the platinum ones. After from twelve to twenty-four hours there appeared at the positive pole an adhering mass. The results were the same as before.

With the nitrate of aniline as the electrolyte there was a black adherent deposit around the positive pole. This became velvety when it came in contact with alkalis. Sulfuric acid decomposed it yielding a brown-red coloration. The hydrochlorate of aniline also yielded a black coloration at the positive pole. However, the results are a bit complicated since there are bubbles of oxygen as well as chlorine present. The tartrate and the acetate of aniline did not give the black. Perhaps if these experiments could be explained in the light of modern atomic theory they might be clarified.

One year before this work of Coquillon, Kruis had finished some experiments on aniline black. He had experimented with manganese, iron and cerium. The black obtained with the use of cerium bisulfate was the best. Cerium was, however, a very expensive chemical element.

Other metals such as uranium, copper, iron and vanadium were tested by Lightfoot in eighteen sixty-nine. From his experiments, he learned that vanadium brought about the desired results more rapidly than copper, uranium less readily than copper and iron the least readily.

A difficulty that apparently was always popping up was, what could be done to achieve a black that would not green. It was the necessity for finding this out that was the spur to so much of the research. During the course of the chemical reactions for the formation of aniline there appeared first the dark green, the emeraldine of Crace, Calvert and Lowe; further oxidation transformed this into a black. This was the nigraniline of Rheineck. Reaction furthering the oxidation formed what was known as aniline black. This was supposedly ungreenable. When an ungreenable black was actually obtained it was of the fastness of the now well-known indanthrene dyestuffs. Such fastness was one of the reasons why logwood and other black dyes had to make way for it. The comparatively low cost of producing it was another reason.

Oxidizing agents besides the chlorate were experimented with. In Germany the purple-colored potassium permanganate was tried. Experiment showed that the addition of potassium permanganate to a sulfuric acid solution of aniline sulfate formed a dark green precipitate. This reacted with alkalis to form aniline black.

The aniline salt largely used was the hydrochloride and was obtained by the reaction of aniline and pure hydrochloric acid. This reaction took place in earthenware or lead-lined vessels. Much fuming and great heat was evolved. Grey crystals of the aniline salt appeared upon cooling the solution. Here great caution must be exercised since the aniline oil fumes are poisonous. The hydrochloric acid has a corrosive effect. This objection was not

(Continued on page 642)

PROCEEDINGS OF THE
American Association of Textile Chemists and Colorists

OCTOBER 18, 1937

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Members should notify **Secretary** immediately of all address changes

Calendar of Coming Events

Meeting, New York Section, Swiss Chalet, Rochelle Park, N. J., October 22, 1937.

Meeting, South Central Section, Farragut Hotel, Knoxville, Tennessee, Oct. 30, 1937.

Fall Meeting, Piedmont Section, Carolina Hotel, Pinehurst, N. C. November 6, 1937.

Annual Meeting and Convention, December 3 and 4, 1937, Bellevue-Stratford Hotel, Philadelphia, Pa.

The Determination of Unsaponifiable Non-Volatile Matter in Sulfonated (Sulfated) Oils

Report No. 7 of the Sub-Committee on Sulfated Oils*

RALPH HART,
Chairman

STRAIGHT sulfonated oils invariably contain a small quantity of unsaponifiable non-volatile organic matter, due to the unsaponifiable matter naturally occurring in the raw oils used for sulfonation. Since sulfonated oils particularly sulfonated olive or olive-type oil are miscible with mineral oils to a large extent, some sulfonated-oil blends may contain as much as 80 per cent of mineral oil. Hence, a reliable method for the determination of unsaponifiable matter in sulfonated oils is a matter of considerable commercial importance.

The first step in determining the unsaponifiable matter in sulfonated oils is to obtain the total de-sulfated fatty matter, which is done by boiling the sample with mineral acid to decompose it, extracting the fatty matter, evaporating the solvent, and drying the residue to constant weight. The problem of determining the unsaponifiable matter in the residue is then identical with its estimation in raw oils. There are two general procedures for determining unsaponifiable matter in raw oils, namely, the Spitz-Hoenig or petroleum ether method and the ethyl ether method. In this report both procedures are first discussed, followed by the recommendations of the Committee.

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Stephen K. Ford, Marden-Wild Co., Somerville, Mass.
Arthur L. Fowler, Jr., American Cyanamid & Chemical Corp., Woodbridge, N. J.
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Ralph Hart, 1440 Broadway, New York City.
Geo. A. Howell, Jr., Rock Hill Printing & Finishing Co., Rock Hill, S. C.
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I. Silverman, L. Sonneborn Sons Inc., Belleville. N. J.
W. S. Williams, Mt. Hope Finishing Co., North Dighton, Mass.

Others Who Have Cooperated on This Report

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*Members of the Sub-committee on sulfonated oils.

PETROLEUM ETHER METHOD

According to the petroleum ether method, the sample is saponified with alcoholic potassium hydroxide, the soap solution made 50 per cent alcoholic, and the unsaponifiable matter extracted with petroleum ether. This method is official for normal animal and vegetable fats and oils with the American Chemical Society[10], American Oil Chemists' Society[10] and the Association of Official Agricultural Chemists.[8]

This method is convenient but unreliable in the case of abnormal fats and oils containing excessive amounts of unsaponifiable matter, higher alcohols, fish liver oils, whale oils, waxes, and other unsaponifiable matter which are insoluble or sparingly soluble in petroleum ether. Even for normal fats and oils the results may be low and erratic. Thus, Jamiesen and McKinney[6] report for the unsaponifiable matter in a mixture of olive and teaseed oils as 0.85 to 1.06 per cent by the petroleum ether method and 1.27 to 1.28 per cent by the ethyl ether method. Jamiesen further states that in some samples of olive oil more than seven extractions with petroleum ether are required in order to get all the unsaponifiable matter. He is also of the opinion that the unsaponifiable matter in olive oil is composed of substances widely different in composition and solubility in petroleum ether. A Committee of the Deutsche Geselschaft für Fettforschung[7], after investigating the two methods, reports that the unsaponifiable matter found in coconut oil by the petroleum ether method was about half as much as obtained by the ethyl ether method.

According to Davidsohn and Better[2] free alkali in the alcoholic soap solution increases the solubility of soap in the petroleum ether layer (although the quantity is negligible) and also increases the solubility of the unsaponifiable matter in the soap solution. As a slight excess of free alkali improves the separation of the layers without much effect on the solubilities and as it also prevents the hydrolysis of the soap, they recommended that the excess alkali after saponification be first neutralized and then 2 cc. of N/2 of KOH solution be added in excess. Since the nature of the unsaponifiable matter is sulfonated oils particularly in bended oils is an unknown quantity, this

Committee does not recommend the petroleum ether method. However for the sake of the records the method as recommended by the Association of Official Agricultural Chemists[8] is given below.

PETROLEUM ETHER PROCEDURE

Petroleum ether.—Redistil below 75°. Make a blank determination by evaporating 350 cc. of the reagent with about 0.25 g of stearine or other hard fat (previously brought to constant weight by heating) and drying as in the actual determination. The blank must not exceed a few mg.

Extraction cylinder.—Glass-stoppered, graduated at 40 cc, 80 cc, and 130 cc, and of the following dimensions: diameter about 1⅜ in., height about 12 in.

Determination.—Weight 5g (±0.020g) of the prepared sample into a 200 cc. Erlenmeyer flask, add 30 cc. of redistilled approximately 95 per cent alcohol (by volume) and 5 cc. of 50 per cent aqueous KOH, and boil the mixture for 1 hour under a reflux condenser. Transfer to the extraction cylinder and wash to the 40 cc. mark with redistilled 95 per cent alcohol. Complete the transfer, first with warm, then with cold H_2O, until the total volume is 80 cc. Rinse the flask with 50 cc. of petroleum ether and add the rinsings to the contents of the cylinder previously cooled to room temp. Shake as vigorously as possible for 1 min. and allow to settle until both layers are clear, when the volume of the upper layer should be about 40 cc. Draw off the petroleum ether layer as closely as possible by means of a slender glass siphon into a separatory funnel of 500 cc. capacity. Repeat the extraction at least 6 more times, using 50 cc. of petroleum ether for each extraction. Wash the combined extracts in the separatory funnel three times with 25 cc. portions of 10 per cent alcohol by volume, shaking vigorously each time. Transfer the petroleum ether extract to a weighed Erlenmeyer flask and distil; or, if desired, evaporate the petroleum ether on a steam bath in a current of air. Heat the flask with residue until a constant weight is obtained in an oven at a uniform temp. not less than 100°. (A vacuum oven may be used at a corresponding temp., which depends upon the pressure used in it. It is important to displace with air any residue vapors of petroleum ether remaining in the flask after heating and before it is weighed.) Deduct any blank from the weight before calculating unsaponifiable matter. Test the final residue for solubility in 50 cc petroleum ether at room temp. Filter, and wash free from the insoluble residue, if any. Evaporate and dry in the same manner as before.

ETHYL ETHER METHOD

In the ethyl ether method the sample is saponified with alcoholic potassium hydroxide in the usual manner, the unsaponifiable matter extracted with ether, and the ether layer washed with alkali and water to remove fatty acids and soap. The procedure is more lengthy and tedious than by extraction with petroleum ether and there is also a tendency for emulsions to form. However, by carefully following the procedure recommended by the Society of Public Analysts (England)[1], the trouble due to emulsions is either entirely overcome or greatly minimized. The method is official for abnormal fats and oils with excessive amounts of unsaponifiable matter, higher alcohols, fish liver, whale oils, waxes, etc. with the American Chemical Society[10], American Oil Chemists' Society[10], Association of Agricultural Chemists[9], Wizoeff (Germany)[18], and the Society of Public Analysts (England)[1].

A sub-committee of the Society of Public Analysts[1] after a number of years of investigation remarks that it is essential to use ether in the determination of unsaponifiable matter in oils and fats, notwithstanding its greater solvent power for soap, rather than petroleum ether. Vesely and Chudozilov[12] state that ethyl ether extraction methods must be employed for materials containing waxy matter as the low results obtained with petroleum ether appear to be due to loss of aliphatic alcohols and sterols. They also remark that 3 extractions of lanoline with ether yielded as much unsaponifiable matter as 6 extractions with petroleum ether. In view of the reports in the literature on the subject, the Committee deemed it advisable to investigate the ether method only and as a result of its findings has proposed this method as a standard; detailed procedure of the method recommended is given at the end of this report.

Precaution to be Observed—The sub-committee of the Society of Public Analysts[1] finds that the most satisfactory solution from which to extract the unsaponifiable matter with ethyl ether is 30 per cent alcohol by volume. It is important to maintain the volume of ether at not less than 150 cc, otherwise small quantities of unsaponifiable matter may be lost. The ether layer is washed with potassium hydroxide in preference to sodium hydroxide because less emulsification takes place and solid fatty acids are more easily removed. According to Grossfeld and Mechlinski[3] an excess of alkali of about 40 cc. of 0.5N of KOH and a boiling period of 15 minutes suffice for the complete saponification of an edible fat.

COMMITTEE RESULTS

Committee samples F and F-1 were submitted to the Committee with instructions to determine in each sample the de-sulfated fatty matter and the unsaponifiable matter —the former by the A.A.T.C.C. method[5] and the latter in accordance with the method given at the end of this report. Sample F was a 75 per cent commercial sulfonated olive oil and Sample F-1, a mixture of 85 parts of the former and 15 parts of Squibb's heavy medicinal mineral oil having a specific gravity of 0.888 at 15.5° C. The de-sulfated fatty matter found by the Committee varied from 64.20 to 64.81 per cent (average 64.55 per cent) for Sample F, and from 69.72 to 70.47 per cent (average 69.67 per cent) for Sample F-1. The agreement of the results on the de-sulfated fatty matter among the members of the

TABLE I

Committee Results of Unsaponifiable Non-volatile Organic Matter in Sample F and Sample F-1

Unsaponifiable Matter

	Sample F				*Sample F-1*			
	I %	*II* %	*Ave.* %	*Deviation from Mean* %	*I* %	*II* %	*Ave.* %	*Deviation from Mean* %
Fowler	....	....	....	....	14.58	14.93	14.75	0.34
Ford	0.71	0.63	0.67	0.07	15.40	15.61	15.50	0.41
Grimshaw-Ogletree	0.63	0.61	0.62	0.02	15.33	14.95	15.14	0.05
Gulick-Segessemann	0.58	0.67	0.63	0.03	15.36	15.59	15.47	0.38
Hart	0.61	0.57	0.59	0.01	15.40	15.25	15.32	0.23
Pingree	0.51	0.64	0.58	0.02	14.71	15.03	14.91*	0.18
Silverman-Mazur	0.58	0.53	0.56	0.04	14.68	14.83	14.76	0.33
Williams-Brown	0.62	0.60	0.61	0.01	14.88	14.99	14.94	0.15
Mean		0.60				15.09		
Calculated		....				15.51		
Greatest Difference		0.11				0.75		
Average deviation		0.03				0.26		

*More than 2 determinations.

Committee was of the same order for each sample as previously reported by the Committee[5]. The results found by the Committee for the unsaponifiable matter are given in Table I. Excellent agreement was obtained for the naturally-occurring unsaponifiable matter in Sample F, namely, greatest difference of 0.11 per cent and average deviation of 0.03 per cent. In the case of Sample F-1, the mean result of the unsaponifiable matter was found to be 15.09 per cent compared with 15.51 per cent by calculation; the difference is probably due to the slight volatility of the mineral oil under the conditions of drying to constant weight. The precision of the result for this sample was of the same order as for the desulfated fatty matter.

COMMENTS AND SUGGESTIONS

The following comments and suggestions were received from the members of the Committee:

R. HART: According to Jamiesen[11] the term "Unsaponifiable Matter" in fat analysis includes all of those substances that are not saponified by alkali, but which are soluble in ether or petroleum ether. As this definition includes also substances volatile below 80° C, the determination in this report is referred to as "Unsaponifiable Non-volatile Matter." In the presence of volatile matter, soluble in solvents, the unsaponifiable matter should be determined after the sample is freed from the former by steam distillation.

Most published methods on the determination of unsaponifiable matter recommend evaporating the solvent at as low a temperature as possible, in some cases at room temperature. To avoid great loss of time, a temperature of 80° C. is specified in the English method. In the presence of substantial amounts of unsaponifiable matter of comparatively high volatility, it might be advisable to dry the total de-sulfated fatty matter in the same manner as the unsaponifiable matter, namely, for 15-minute periods at 80° C.

R. A. PINGREE: As a result of considerable work done in this laboratory on the solubility of oils, fats and waxes in various solvents, I suggest that, instead of ethyl ether or petroleum ether being used for extracting unsaponifiable matter, we use a mixture of the two containing 4 parts of petroleum ether and 1 part ethyl ether by volume. This mixture is an excellent solvent for all unsaponifiable oils and waxes and is not a soap or sulfated oil solvent to any appreciable extent. For these reasons, separations are more rapid and clean cut.

In drying the de-sulfated fatty matter I believe that the same temperature should be maintained as when drying the unsaponifiable matter. If the unsaponifiable is volatile to any extent, then some of it might be lost in drying the de-sulfated fatty matter.

SEGESSEMANN: In saponifying the fatty matter with alcoholic KOH, we found it necessary to use a water-cooled reflux condenser to prevent excessive loss of alcohol. In all other respects the analyses were carried out strictly in accordance with the instructions. The unsaponifiable residue in each case was found to be entirely free from fatty acids when tested in accordance with the instructions.

For the natural unsaponifiable matter of fatty oils and for instances where the unsaponifiable consists of heavy mineral oil, a drying temperature ranging from 100° to 105° C. seems to me the most satisfactory. For sulfated oils, however, which may contain lighter mineral oils, ranging from low-viscosity white oils to mineral seal oil, a temperature of 100° C. would, of course,

be too high, and even at 80° C. appreciable losses would have to be expected. Furthermore, some of the high molecular alcohols which are encountered with ever increasing frequency in the sulfated products on the market are partly volatile at or even below 100° C. For the present, I would suggest that the drying temperature be specified at 100-105° C. and that the designation of the method be changed to read "determination of non-volatile unsaponifiable matter."

SILVERMAN: While this method appears to be rather lengthy with considerable cross washing, we do not believe it is cumbersome or difficult. This determination should really be referred to as "Water Insoluble Unsaponifiable Matter" in view of the fact that glycerine, glycol, etc., would not be included in this unsaponifiable matter?

Proposed Standard Method of Test for Unsaponifiable Non-Volatile Matter in Sulfonated (Sulfated) Oils

Scope:

1. This method of analysis determines the unsaponifiable non-volatile (below 80° C.) matter existing in a sample of sulfated oil by saponifying the de-sulfated fatty matter and extracting the unsaponifiable matter from the soap solution with ethyl ether.

Apparatus:

2. *Saponification Flask*—The apparatus required for the saponification consists of a glass flask provided with an air condenser and identical with the apparatus described in the A.A.T.C.C. method for organically combined sulfuric anhydride[4].

Note:—To avoid loss of alcohol, a water-cooled condenser may be substituted for the air condenser.

3. (a) *Potassium Hydroxide Solution* (0.5*N*)—Prepare an approximately 0.5 *N* aqueous KOH solution.

(b) *Alcoholic Potassium Hydroxide* (0.5*N*)—Prepare an approximately but not less than 0.5 *N* alcoholic KOH soultion.

(c) Ethyl Ether, U.S.P.

Procedure:

4. The procedure consists in decomposing the sample with mineral acid, extracting the de-sulfated fatty matter, saponifying the latter, and extracting the unsaponifiable matter from the soap solution with ethyl ether.

(a) *De-Sulfated Fatty Matter*—Determine the de-sulfated fatty matter according to the A.A.T.C.C. method for total de-sulfated fatty matter[5].

Note: The de-sulfated fatty matter should be heated to constant weight preferably at 75° to 80° C, particularly if unsaponifiable matter volatile above 80° C. is suspected.

(b) *Saponification*—Accurately weigh 2 to 2.5 g. of the de-sulfated fatty matter in the flask, add 25 ml. of the alcoholic KOH, and simmer the contents (without loss of alcohol) for 1 hr. with occasional swirling over an electric hot plate or other sources of heat.

(c) *Extraction*—Transfer the contents of the flask to a 250-ml. separatory funnel and wash the flask several times with a total of 50 ml. of water, pouring it into the separatory funnel. Extract the solution, while still warm (about 30° C.), with 50 ml. of ether (first rinsing the saponification flask with the ether before adding to the separatory funnel), shake vigorously for 1 minute, and allow the layers to settle and clear. Draw off the lower layer in a second 250-ml. separatory funnel and extract in a similar manner with two 50-ml. portions of ether.

(d) *Preliminary Water Washing*—Add 20 ml. of water to the combined ether layer, turn the contents over gently about six times, and allow the layers to settle and clear. Draw off the lower layer and discard. Wash the ether layer twice more with 20-ml. portions of water, shaking vigorously after each addition.

(e) *Alkai and Water Washing*—Add 20 ml. of 0.5 *N* KOH to the washed ether layer, turn contents over gently about six times, and allow the layers to settle and clear. Draw off the lower layer and discard. Wash the ether layer with 20 ml. of water, shaking vigorously. Continue washing with 20-ml. portions of 0.5 *N* KOH followed by 20-ml. portions of water, shaking vigorously after each addition, until the akali layer upon strong acidification with HCl and standing for a few minutes is only faintly opalescent. Wash the ether layer with 20-ml. portions of water until the wash water is no longer pink to phenolphthalein.

(f) *Solvent Removal*—Transfer the ether layer to a 150-ml. beaker (counter-balance the beaker if the amount of unsaponifiable matter is small and important) and evaporate over a hot-water bath until practically free of ether. Heat the beaker at 75° to 80° C. until constant weight is obtained.

Note 1—A 30 per cent alcoholic solution by volume is the most satisfactory solution from which to extract the unsaponifiable matter.

Note 2—If emulsions form, add 5 ml. of alcohol, pouring it down the side of the funnel.

Note 3—It is important to maintain the volume of ether at not less than 150 ml., otherwise small quantities of unsaponifiable matter may be lost.

Note 4—To check whether or not the unsaponifiable matter is free from fatty acids, dissolve the residue in 10 ml. of freshly boiled and neutralized alcohol and titrate with 1/10 *N* alcoholic potassium hydroxide solution, using phenolphthalein as the indicator. Not more than 0.1 ml. should be required for neutralization. If more is required, the determination has not been carried out effectively and must be repeated.

Calculation:

5. Calculate the unsaponifiable matter as follows:

$$\text{Unsaponifiable non-volatile matter, per cent} = \frac{\text{Weight of residue in grams}}{\text{Weight of de-sulfated fatty matter in grams}}$$

times percentage of de-sulfated fatty matter.

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MEMBERSHIP APPLICATIONS

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C. Harrell Asbury — Demonstrator, E. I. du Pont de Nemours & Co., Inc., Charlotte, N. C. Sponsors: H. G. Miller, L. W. Sherrill.

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MEETING, PHILADELPHIA TEXTILE SCHOOL STUDENT SECTION

THE first meeting of the current year of the Philadelphia Textile School Student Section was held on Monday, September 20, at 4:00 P.M. with Chairman Wayne Fry presiding. The freshman class was welcomed and activities of the Section were explained to them.

Several of the members have volunteered to relate their experiences in the industrial world during the summer to the Section, and they will be given the entire next meeting period for this purpose.

Respectfully submitted,

ROBERT F. ROECKER, *Secretary.*

COMMUNICATION TO MEMBERS

Dear Average Member:

PROGRAM FOR ANNUAL MEETING

ON December 3 and 4 the American Association of Textile Chemists and Colorists will hold its Seventeenth Annual Meeting in Philadelphia. The advantages of attending this meeting will be presented to our members many times before December at the meetings of the local sections and through the pages of the American Dyestuff Reporter. In this letter to the average member I want to make a special appeal on behalf of the Technical Program Committee for greater volunteer participation by our members in the technical program. At our meetings in the past we have had to depend practically entirely upon invited papers, whereas, in the American Chemical Society and in other technical organizations, programs are made up from papers offered voluntarily and without solicitation.

Not only should there be more voluntary papers for our programs, but there should be more papers from practical dyers and chemists in the mills. Our programs have often been severely criticized because most of our speakers have been representatives of chemical and dyestuff firms with something to sell. In fairness to these manufacturers, however, it must be pointed out that for the most part the papers furnished by them have contained original and technical material rather than mere advertising copy. But we do need the participation of more mill men in our programs and since there is little likelihood that the program committee can guess who among the hundreds of mill chemists and colorists might have material for a paper, the mill man must volunteer his services.

Come forward mill men and volunteer to do your share for the technical program!

Sincerely yours for a better and more representative program,

CARL Z. DRAVES,
Chairman Technical Program Committee for Seventeenth Annual Meeting.

Notice To Speakers

Technical Program 1937 Annual Meeting of American Association of Textile Chemists and Colorists at Bellevue-Stratford Hotel, Philadelphia, Pa., December 3 and 4, 1937

I. Prospective speakers for the 1937 Annual Meeting of the American Association of Textile Chemists and Colorists in Philadelphia on December 3 and 4 are asked to send the following information to the Technical Program Committee as soon as possible:

a. Title of paper.
b. Name of author (or authors).
c. Firm or laboratory to be credited.
d. Necessity for projection lantern or other special facilities.
e. Time required for delivery of paper.

In order to ensure a place on the program, answers to this notice must not be delayed.

Since we always have very full programs at annual meetings, the committee will appreciate a careful estimate of the time required for the delivery of each paper. The essential material of most scientific and technical papers can be made understandable to an audience in 15 to 25 minutes.

II. An abstract of less than 350 words must be mailed to the Program Committee before November 7. The purpose of the abstract is to give: (1) A more definite description of the nature and scope of the paper than can be conveyed in the title, (2) The essential results insofar as may be possible in the limited space allowed.

It is hoped that the advance publication of these abstracts in the AMERICAN DYESTUFF REPORTER will prepare those attending the meeting to consider the papers more intelligently and with much greater interest. Authors are urged to cooperate in this effort by preparing their abstracts carefully with this end in view.

III. Manuscripts ready for publication must be turned over to the stenographer immediately after presentation. Papers presented before our meetings become the property of the Association as far as publication is concerned and will be published in the Proceedings in the AMERICAN DYESSTUFF REPORTER. When an author so wishes, it is possible to arrange for simultaneous publication in some other technical or scientific journal. Proof sheets of papers and of the discussions following papers will be mailed to speakers for correction before publication.

IV. The Technical Program at Philadelphia will be run off strictly according to schedule exactly as have previous programs. In order that speakers may finish in the time allotted and still leave several minutes for discussion, it is suggested that papers for publication be considerably abbreviated for oral presentation. A listening audience is primarily interested in basic principles, bare outlines of experimental methods and broad conclusions. Important data can well be visualized with large charts or graphs or by means of lantern slides. Mimeographed sheets are sometimes useful. Complete details can then be left for the published article.

V. Programs giving a complete schedule of the time allotted to each paper will be mailed to speakers well before December 3.

The Program Committee wishes especially to thank those who cooperate with us in working for a successful technical program.

CARL Z. DRAVES, *Chairman,*
Technical Program Committee,
273 Converse St., Longmeadow, Mass.

PROGRAM

Fall Meeting of South Central Section

Farragut Hotel, Knoxville, Tennessee, October 30, 1937

2:00 P.M., Football Game, Shields-Watkins Field

UNIVERSITY OF GEORGIA

vs.

UNIVERSITY OF TENNESSEE

West Stand $2.50: Tax Included

East Stand $2.00: Tax Included

Tickets may be obtained by sending remittance and postage to either:

Mr. Robert Cowan, Industrial Colloids and Chemical Co., Knoxville, Tennessee.

Mr. William S. McNabb, Quaker City Chemical Co., Knoxville, Tennessee.

7:30 P.M., Banquet, Assembly Room, Farragut Hotel

Address of Welcome, City Manager, Mr. George Dempster

8:00 P. M., Business Meeting

Chairman will name Nominating Committee to consider Nomination of Officers, for year 1938; Committee to report at January Meeting.

8:15 P. M., Technical Program

1. Paper by Mr. Nestor Grotelueschen
2. Paper by Mr. Walter E. Hadley

Farragut Hotel is kindly reserving a limited number of rooms for guests at this meeting, and it is suggested that early reservations be made direct with the hotel.

ANNUAL MEETING AND CONVENTION

COMMITTEES

ELMER C. BERTOLET—General Chairman
Alban Eavenson—Vice-Chairman
William A. Stringfellow—Treasurer
Richard M. Ritter—Secretary

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Robert O. Beadel Robert W. Delaney
Harold B. Dohner

FRED SCHOLLER
Member, Entertainment Committee

ENTERTAINMENT

Reports from Arthur E. Jones, Jr., chairman of the entertainment committee, indicate that the program of entertainment this year will be of the usual high standard characteristic of the annual meetings of the Association. A well known orchestra has already been engaged and negotiations are now under way to provide additional entertainment.

ARTHUR E. JONES, JR.
Chairman, Entertainment Committee

DECEMBER 3rd and 4th PHILADELPHIA, PA.

AMERICAN
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REPORTER

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THE CONVENTION CITY

PHILADELPHIA, where the Annual Meeting of the American Association of Textile Chemists and Colorists will convene on December 3rd and 4th, is one of the most important textile centers of the country and thus is especially well suited for a convention of this sort.

According to the *Evening Bulletin*, Philadelphia newspaper, the textile industry is the largest of any in the city and its importance is felt by everyone of the city's population of 2,000,000. The *Bulletin* presents the following facts to substantiate its statement. In the past five years this industry has been producing more and more products and employing a larger and larger number of people. In 1936, sixty-three new textile firms located in Philadelphia. Over $143,000,000 of capital is invested in Philadelphia alone; normally textiles employ nearly 95,000 Philadelphian workers, and the value of textile products produced yearly is normally $493,000,000. Philadelphia manufactured more than one billion, two hundred million pairs of hosiery in 1935; nearly six million, five hundred thousand shirts; over four million hats and caps; almost five hundred thousand pairs of overalls and many more than three hundred thousand handkerchiefs. Thirty-seven and nine-tenths per cent of Philadelphia's industrial wage earners are employed in the textile industries and receive twenty-nine and four-tenths per cent of all wages paid and produce twenty-one and six-tenths per cent of the value of products made in all of Philadelphia's industrial establishments.

A break-down of these figures discloses that on the basis of value of products, knit goods, with 16 per cent of the establishments, is the most important branch, accounting annually for 25 per cent of the value of all textile production. Men's and boys' clothing and women's clothing are second and third in this respect, accounting for 22 per cent and 20 per cent of the textile production respectively. In number of wage earners and wages paid, the same relationship exists; knit goods are most important with 27 per cent of the wage earners and paying 33 per cent of the wages paid in the textile industry.

In addition to its textile importance, Philadelphia is rated the third foremost manufacturing city in the nation and also the third largest commercial center. Its manufacturing industries produce $283,000,000 worth of manufactured products annually; its 30,000 retail stores sell $657,000,000 worth of goods each year; its more than 3,900 wholesale establishments sell annually $1,293,000,000 worth of merchandise and its 1,300 service establishments collect an additional $53,000,000 yearly in receipts.

Historically, Philadelphia has much to offer the visitor. Among the places about which we all have heard and which can be seen are Independence Hall, Congress Hall, Carpenter's Hall, the oldest bank building in the country, the famous Chew House, Christ Church burial grounds, Betsy Ross House, Penn Treaty Park and Valley Forge just a short distance out of Philadelphia.

All in all, Philadelphia offers historically, commercially and industrially a fine city for the

ANNUAL MEETING
AND CONVENTION

IMPORTANT NOTICE!

The Next
QUARTERLY
RESEARCH NUMBER

originally scheduled for this issue will appear in our issue of

NOVEMBER 1, 1937

AMERICAN DYESTUFF REPORTER

Historical Review of
Aniline Black

(Continued from page 632)

overcome until nineteen fourteen. During that year two chemists, Drs. Conway and Girsewald perfected a method for the making of aniline salt by the use of iron and a catalyzer, ferrous chloride.

The black obtained by the use of the permanganate was comparable with that produced by the copper sulfide. Cerium black, though expensive, was still considered the most satisfactory of all. Vanadium black was applied by Guyard and Witz to furniture cloth. It was also used in Germany for dyeing wool and silk as well as leather. Another use for it was in the making of black varnishes since it was soluble in alcohol. Its alcoholic solution was merely mixed with the varnish-producing oils and resins. To dye wool vanadium aniline black was used. It was found that feathers could be dyed directly with aniline black.

It was during this year of eighteen seventy-six that much litigation ensued when patents on aniline black were granted to Samuel Grawitz in France. It was claimed that these patents infringed upon the rights of others and that there was nothing novel in them. Furthermore, such patents should never have been granted.

The challenging and most compelling question of how the aniline black dyestuff was formed was answered by Rosenthiel, experimentally, in eighteen seventy-six. He showed that the reaction is entirely due to the compounds of chlorine and oxygen. Metal need take no part in the reaction.

This year, too, brought forth the chemical formula of aniline black. Ever since Fritsche's fruitless attempts at discovering it, numerous investigators had obtained a few facts concerning it. These culminated in Nietzki's announcement that aniline black was a polymer of a unique unit grouping of atoms. These were carbon, hydrogen and nitrogen. Six atoms of carbon, five of hydrogen and one of nitrogen. It was written $(C_6H_5N)_x$. The unit might be thought of as a molecule of aniline from which two atoms of hydrogen had been removed. Further work on this Herculean task was not completed until the end of the first decade of the twentieth century.

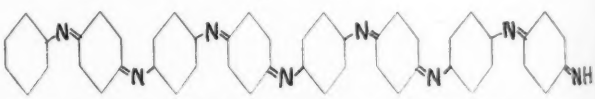
Gradually the superiority of the prussiate method for the dyeing of aniline black was recognized. In this the fiber is tendered the least since the hydrochloric acid can be completely neutralized. The vanadium black was also used since the salts of vanadium had become cheaper. With the proper technique the copper sulfide black gave good results. The lead chromate black, introduced by Miggins in eighteen sixty-six when he showed that lead chromate rendered the black ungreenable, also gained a place for itself in the industrial world.

The efforts made to perfect these four methods stimulated research in the field of inorganic chemistry by causing a demand for better and cheaper oxidizing agents. It was found that barium chlorate could be used in prussiate black printing pastes. It could not be utilized if sulfates were present since the barium sulfate will then precipitate, dulling the shade. Sodium chlorate made in a highly purified state also facilitated the production of the black. The potassium chlorate that played such an important role at first was replaced by these more soluble and cheaper chlorates. Sometimes chloric acid in solution was used.

Research had also included the basic bodies of the aromatic series. In nineteen two, Ulrich and Fussanger completed the work on these chemical compounds. By oxidation of the derivatives of diphenylamine an intense, absolutely ungreenable black was obtained on the fiber. The color developed during the drying of the fabric. A catalytic agent was not needed. This was patented in Germany and placed on the market as Diphenyl Black base. It was, however, more expensive. It was not as fast to chlorine as the other blacks.

In nineteen four aniline black was being used for topping other blacks. It topped Columbia Black B and EB as well as Zambesi Black D. Back in the eighteen hundreds an opposite effect was produced when the aniline black was topped with aniline violet in order to make an ungreenable, aniline black.

The work of Willstaetter and co-workers now revealed that the greenable black, i.e., the nigraniline, was a highly complex induline. The carbon, hydrogen and nitrogen atoms of the polmerized substance were oriented in space according to the following formula:

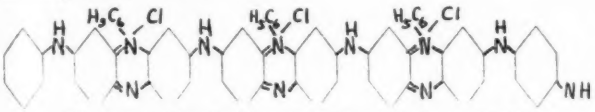


Greenable Aniline Black or Nigraniline

Experiment also brought forth the fact that the compound having the above formula was the aniline black obtained when the single bath was used. By using sulfurous acid this black could be changed into the dark green substance, emeraldine, which, it is thought, is never pure but always contains some nigraniline. The latter gives it the black reflex.

This nigraniline can be oxidized with chromic acid. The result is the ungreenable aniline black, produced by utilization of two baths. Whether this is homogeneous or not has not been definitely decided. Hence the formula offered is not the last word. Although it is conceded that it gives a certain amount of insight into the chemical nature of aniline black. By the aid of bromoaniline, Green

and his co-workers soon showed that three aniline molecules became attached to the nigraniline to form the azine or ungreenable aniline black.



Ungreenable Aniline Black or Azine Black

Whether the nigraniline or the azine black will ever be isolated is a matter of conjecture. Until then the above formulas are a step nearer the light.

Soon after the war, the introduction of the fast color salts, the Rapidogens and the Indigosols necessitated slight modifications in the aniline black processes when it was to be used in conjunction with them.

In an effort to cheapen the aniline black processes, those chemicals that were most injurious to the fibers were left out. Here however the results are not as ideal as could be wished for. It is said that this black is not ungreenable. About a decade later in 1936 this Eumol process was tried again. Whether with any greater success was not reported.

About nineteen twenty-eight the Russians used a method whereby the amount of aniline oil was increased. This neutralized the acid produced during the steaming process and decreased the tendering of the fiber.

The neutralization of the acid was also brought about by a product placed on the market in Germany in nineteen twenty-eight. This was known as "Kollamin". It reacted by combining with the excess mineral acids and made the oxygen of the oxidizing agent ineffective. An absolutely ungreenable black is claimed to be produced by this method. It is particularly useful with resists since the black requires a longer time to form. Another advantage of making aniline black this way is that it can readily be applied to half-silk as well as silks.

By nineteen thirty-four Rapidogens were being used with aniline black with zinc oxide acting as the resist. The oxide was mixed with the dyestuff and on steaming the color developed. The prussiate aniline black liquor is then padded in. Upon drying and steaming, the black develops with the zinc oxide acting as the resist. Vat colors such as the indigoid dyestuffs and the indanthrenes can also be used with aniline black provided zinc oxide is added to the colored dyestuff to act as a resist. Insoluble azo dyes on naphthol prepares find uses with aniline black.

It has been recorded that phenol-formaldehyde resins, particularly when it is necessary to be careful about the electrical properties of the substance, have recourse to aniline black.

Whether new uses for aniline black will be developed, or its chemical constitution clarified still further depends on future experiment.

Stainless Steel

Drying Cylinders

SAUL BLICKMAN

STAINLESS steel, the modern material of industry is fast taking its place for use on textile cylinder drying equipment. Wherever used, it is claimed to have given superior satisfaction from the standpoint of maintenance, rapid drying, and general efficiency, and is rapidly replacing older and inferior materials. This comparatively new material is particularly adaptable to the manufacture of drying cans. Modern welding methods have made it possible to fabricate it into cylinders of virtually seamless construction, these seams being so carefully and neatly made as to be nondiscernible to the eye. The experienced fabricator can produce a stainless steel drying can to meet the demands of the most exacting mill operator or dye house man. The seams are welded in a manner to assure greater strength at the joint than in the adjoining material. (In tension this metal is "good for" 90,000 lbs. per square inch). Modern manufacturing methods have made it possible to finish the welded seam on the outside so that it is absolutely continuous with the rest of the cylinder; to make the joint invisible; to have a continuous rounded effect adjacent to the joint. Any well made dry can should include all of these features, namely, the cylinder should be round, (there should not be a variation of more than 3/64" in the trueness of diameter) the weld and the adjacent material should have no flats, and the seam should be invisible and polished to match the surrounding material. A cylinder having these features is insurance against spoilage of goods during drying processes.

An all welded seamless steel cylinder, 16 gauge, costing but little more than a copper cylinder will outlast a copper cylinder 10 times. In fact, it is fair to claim that the life of a stainless steel cylinder is indefinite, as such drying cans are impervious to the liquors with which they come in contact. This property, coupled with the toughness of the material and the ability of fabricators to increase the

corrosion-resistant properties by putting on high finishes, makes the installation of the stainless steel cyinder a permanent one. Stainless steel has a tensile strength 10 times that of copper and a resistance to abrasion many times that of copper. These properties assure against cylinders "blowing up" or collapsing and against scouring and pitting.

Due to the toughness of the material and its resistance against corrosion, the servicing of stainless steel cylinders has been reduced to a negligible item. With them there are no costly retinning jobs, no losing the services of the machine because cylinders must be sent out to be replated or retinned. Should they become damaged through abuse, they can readily be repaired. The high elasticity of the metal makes it possible to remove pieces several square inches in area and weld in their place undamaged pieces. This type of repair gives to the user a cylinder that is just as good as new. The welded-in piece can be welded in in such a fashion as to be absolutely continuous with the undamaged portion of the cylinder, at the same time being steam tight.

Due to the relative thinness of material which is used for stainless steel cylinders, its heat transfer is very high. Copper cylinders become badly corroded on the interior and soon a film of oxide forms on the inside of the cylinder due to the action of steam in attacking the copper. This film defeats the effect of the high conductivity of the copper by slowing up the heat transfer from the steam to and through the copper. Stainless steel, not being subject to such attack by the action of steam, does not corrode and there is an immediate heat transfer to and through the metal. To the knowledge of the writer there has never been a single replacement of copper cylinders by stainless steel cylinders where any noticeable difference has been observed due to the relative variation in conductivity of the two materials. At the same rate of speed of material over the cylinders, whether of stainless steel or copper, the same drying effect was noted. In other words, to do a given drying job, it can be done just as readily and rapidly with the same number of stainless steel cylinders operating at the same rate of speed as if copper cylinders were used. The question of relative conductivity does not appear to enter at all. What counts is that sufficient heat is transferred during the time of contact of the material to give the drying result required.

A condition greatly affecting the heat transfer through a cylinder wall is the formation of an oxide on the exterior of the cylinder. The formation of even a slight oxide will to a very large extent retard the transfer of heat to the material to be dried. Copper tarnishes very readily forming an oxide where exposed to the air. Stainless steel retains its bright lustrous polish and does not form such an oxide and has a distinct advantage in this respect over copper.

The plating of a film of chromium over a soft copper cylinder has failed to meet the requirements of service. The plating of a film of chromium is recognized in industry as producing hardness and resistance to wear. The effect of this chromium plating has not been fully beneficial for the reason that the very thin film of chromium is plated over a soft copper base and the chromium film does not in any way add to the toughness, the resistance to bursting or collapse of the copper cylinder, as basically a soft copper cylinder has not sufficiently high physical properties even with a chromium film plated over the soft copper cylinder.

It would appear that the substitution of stainless steel drying cylinders for copper, as well as for chromium plated copper cylinders will effect a great economy in operation and maintenance. The cost of such cylinders, while slightly higher than those of copper, will afford savings in operation and maintenance which far outweigh the difference in first cost. The cost is also considerably less than that of chromium plated copper cylinders and not only is a saving in first cost effected, but their use assures absolute permanence and is insurance against replacement for many years.

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Textile Institute Conference

Southport, England, June 9-11, 1937

Discussion on Serviceability of Fabrics in Regard to Wear—Reported in Part

(Concluded from October 4th issue)

Abrasives for Wear Testing

J. H. Lester, M.Sc., F.I.C., F.T.I.

WHEN two portions of a uniform fabric are rubbed together, it may be said that the abrasive is definitely

standardized, but the value of such "cloth-on-cloth" tests as an indication of the life of a fabric in actual use has yet to be explored or determined. Such tests may easily be made by the "Herzog-Geiger" or the "Ring-Wear" method.

There is a tendency to-day to discard the use of carborundum or other abrasives which are supposed to have a severe cutting action upon fibers, though it must be admitted that if a number of different kinds of fabric are tested with carborundum and also with a softer abrasive, they are often found to range themselves in the same order of merit.

That the nature of the abrasive not only affects the rapidity but the essential character of the results is shown in a very marked degree when fabrics of cotton and of spun viscose are compared:

Abrasive	*Cotton*	*Viscose*
Card Clothing	263	635
Ring-wool	2880	830

The reversed superiority of the two fabrics according to the abrasive employed clearly indicates that essentially different physical properties are measured by the different abrasives. The Amsler type of machine attempts to eliminate surface friction as far as possible and to produce the maximum of friction between fibers throughout the fabric, by the use of smooth steel rods which flex the whole structure; but surface friction still plays some part. Experience with the use of card-clothing, whereby a brush of steel wires is "trailed" over the surface of the fabric, shows that although the effect is largely one of surface abrasion, it results in much teasing out of the fibers, provided, of course, that they are not too hard or brittle to be withdrawn from the fabric.

This experience clearly suggests that one factor in the resistance of a fabric to wear is the resistance of the fibers to forcible withdrawal from the fabric and at the same time their resistance to breakage during withdrawal.

The tendency of some fabrics to develop a "raised" surface or tufts or neps during testing and during wear is certainly one which needs investigation. The selection of a suitable abrasive is only one of a number of factors which must receive attention. Whatever the abrasive, we cannot overlook the presence of the broken fibers we always produce, in themselves a very disturbing abrasive. It would be out of place here to deal with many other disturbing factors, such as the presence of small amounts of lubricating substance in the fabric; the moisture content and the effect of the heat generated by friction upon that content; standardization of the tension of specimens under test and of the load to be applied, etc.

New Dyes and Finishes

H. B. Bradley, B.Sc.

THE flax fiber has inherent properties which have given linen fabrics a reputation for genuine value and beauty, and this should be kept in mind by all in the linen section. New products of the manufacturers of dyestuffs, auxiliary products, and textile machinery should be exploited to their fullest advantage in maintaining and furthering the attractiveness combined with real wearing qualities and serviceability.

There is now an extensive range of dyestuffs of good all-round fastness available, and it is regrettable that acute price-cutting should prevent their fuller use. Attractive and permanent finishing effects by various new methods and treatments are capable of enhancing the selling value and quality of linen, and would bring great benefits to the manufacturers of dress materials.

One of the greatest difficulties in linen dyeing is the problem of obtaining fully penetrated dyeings, particularly when using the faster vat dyestuffs. In the case of the insoluble azoic dyestuffs produced on the fiber, penetration is not such a serious difficulty. Soluble derivatives of the leuco-compounds of vat dyestuffs give good penetration but can only be used at present in the production of pale shades on account of their very high cost of manufacture. Vat dyeing has lately been facilitated by the introduction of methods and machinery for forcing the dye while in the unreduced state through the fabric by mechanical pressure, followed by reduction and fixation of the dye as a distinct stage of the dyeing process.

Launderability of Fabrics

F. Courtney Harwood

THE laundry quite often receives more than its fair share of blame in connection with the failure of textiles. Many faults and damages only become apparent on laundering, and frequently it is not possible to allocate the responsibility for these. Then the launderer has to choose between losing a customer and paying up.

It is regrettable that the term "unshrinkable" is applied indiscriminately to all kinds of woolen underwear, quite apart from any consideration of whether the fabric has received an adequate unshrinkable finish and apart from any consideration of fabric structure. Of course, in the textile world it is realized that at best "unshrinkable" (like "fadeless" or "fast color") is a relative term, but a large proportion of the general public certainly does not.

It is possible to wash woolens which have received adequate chlorination in a rotary machine quite satisfactorily from the point of view of shrinkage, providing suitable precautions are taken, particularly with reference to the amount of mechanical motion received. When many so-called unshrinkable woolens are washed by such a process the result varies from the merely unsatisfactory to the extremely bad. You may say these garments give satisfactory service if they are washed according to the instructions which are supplied by some manufacturers. They will stand up to a gentle hand-washing process, but what is the use of basing a statement of unshrinkability on a washing process which is frequently incapable of effecting adequate cleansing?

Mr. T. C. Petrie said in connection with laundering: There is also the important problem of the designer. At times it becomes painfully evident that a straining after effect gives rise to much trouble in wear and in laundering. For example, in high quality silk pyjama fabrics, with long floats of silk yarn of low twist, the silk yarn simply rubs out in wear, and if washed, the yarn loses its con-

tinuity. Shirting fabrics of high quality are frequently encountered where the fabric is quite suitable for its purpose, but the color to match the shirt is not, owing to the fact (1) that the collar has a fold, and (2) the warp yarns are running the length of the collar. In the first case, the weft running over the edge of the fold is subjected to the strains and stresses set up by the thickness of the fold. In the second place, it receives the rub not only of the neck itself, but also that of the inside of the coat collar.

Again, does the designer ever consider the effect of wetting and the inevitable contraction which takes place? Take, for example, a recent type of sheet, where a thick cord takes the place of hemstitching. The latter was bad enough, but the cord idea is worse. Such a cord, or rib, on wetting, contracts, stresses and strains are set up, the warps are distorted, and very often so are the wefts. Such an article should be calendered, but the presence of a cord causes much trouble, first of all, to straighten the piece, then having done so, by exerting a good deal of strain without real avail, the piece is calendered, with the result that a dried, wrinkled, trenchlike line runs parallel to the cord, or rib, and the selvedges are pulled out of alignment. Ribbed fabrics, such as shirtings, always split parallel to the ribs, the wefts reach their breaking strain due to the stresses set up by the contraction of the relatively heavy yarn producing the rib.

A great service can be done for all concerned if only the manufacturers and the laundry industry put their minds to closer co-operation.

TRADE NOTES — NEW PRODUCTS

• TERGITOL

The old comparison "like water off a duck's back," will no longer be appropriate if minute traces of a newly developed chemical are added to water, it was announced by Dr. B. G. Wilkes of the Mellon Institute, Pittsburgh, and Dr. J. N. Wichert of Carbide and Carbon Chemicals Corporation, South Charleston, West Virginia, at the recent Rochester meeting of the American Chemical Society. As a result of investigations in the field of synthetic organic chemistry, hitherto unobtainable alcohols of high molecular weight have been made available in quantity sufficiently great to establish them as commercially valuable products. These alcohols, as a series, range in carbon content from ten to twenty-one carbon atoms per molecule and are therefore comparable in molecular size to those alcohols which are now derived chemically from such naturally occurring raw materials as vegetable and animal fats and waxes.

Because of their giant molecular size, these synthetic alcohols are not soluble in water. It is stated however, that by sulfonation, they can be converted into products which are readily dissolved in water and which are said to impart to such aqueous solutions very pronounced properties of detergency, emulsifying action, penetration and wetting-out of large surface areas. It is claimed that a tenth of an ounce added to a gallon of water makes the water actually "wetter," that is, the water penetrates whatever it touches almost instantly. These compounds have recently been made available to industry under the trade-mark "Tergitol."

It is claimed that further investigation of the properties possessed by the "Tergitol" wetting agents shows them to be very effective and quite stable under most commercial concentrations of alkaline, acid, oxidizing, and hard water conditions so frequently met with in textile processes. Even in peroxide and hypochlorite bleaching as well as in mercerizing baths their efficiency is said to be retained undiminished for many days. According to the manufacturers, they are well suited for the impregnation of textile materials where it is desired to impart future absorbency and they have been found to serve as emulsifying agents. It is stated that the "Tergitol" penetrants also aid in the dispersion of lime soaps, thereby reducing the tendency of soaps to form curds in hard water.

The effectiveness of the "Tergitol" penetrants in promoting wetting textile fibers is well illustrated by the fact that, while yarn or thread made from raw or chemically untreated cotton remains unwetted by water for an indefinitely long period of time, it is claimed that cotton in the same state becomes thoroughly wetted in a few seconds by water solutions containing as little as one-tenth of one per cent of these synthetic alcohol sulfates.

Many profitable applications of this new synthetic alcohol sulfate are said to be presented in the textile industry, since most of the operations involve the use of aqueous solutions. It is stated that improved processing results and reduced operating time are realized by employing these "Tergitol" wetting agents in the kier-boiling, bleaching, mercerizing, dyeing and conditioning of cotton yarn and fabrics and in the dyeing, carbonizing, scouring and felting of wool. The manufacturers claim that further variety and volume of usage of the "Tergitol" wetting agents will develop as textile manufacturers become increasingly conscious of the value of "wetter" water.

• DRY FULLING MACHINE

This machine has recently been put on the market by Riggs & Lombard, Inc. of Lowell, Mass. Fully protected by patent applications it is said to be the first machine of its type and has been in successful operation in several well-known woolen mills for a period of two years. It is designed to perform in a continuous manner the operation known in a woolen mill as "dusting" or "crushing", which was formerly carried out on old, rebuilt fulling mills on the batch system. Its continuous operation feature is said to result in greatly increased production.

Fundamentally the machine is a multiplicity of squeeze rolls, the number of rolls being determined by the work to be handled. The cloth is threaded between the rolls in such a manner as to give the maximum number of nips and the number of nips is further increased by an alternating forward and backward movement of the rolls. The ratio between these forward and backward movements is adjustable so as to give delivery speeds of 6 to 30 yards per minute.

The machine can be hooked up in tandem with the carbonizing range, making the processes of soaping, drying, baking and crushing one continuous operation. Even fabrics which are exceptionally difficult to crush or dust are said to be easily handled on this machine, as the number of nips given to the cloth is under the instant, convenient, fingertip control of the operative.

• STAINLESS DYE KETTLES

Birch Bros., Inc., 32 Kent Street, Somerville, Mass., announce production of a line of stainless steel dye kettles consisting of open kettles and enclosed and insulated kettles for either rope or open width dyeing.

The metal used in the construction is said to be of sufficiently heavy gauge and the tub to be entirely independent of the side frames. Bearings, doffing reel arms and motor drive brackets are all supported by the heavy carefully machined side frames, eliminating strains on the tub. The tub is designed to prevent goods packing and tangling.

It is stated that the reels are of unique design made to prevent slippage of the cloth, with the heads or flanges having a long bearing on the shaft and keyed to it. The reel is so made that it may readily be made elliptical or round or any number of rolls may be used. The reel shaft and the doffing reel run in ball bearings.

• GENERAL RELEASES

General Dyestuff Corp., 435 Hudson St., New York City, announces release of circulars on the following new products:

Ramasit K N Conc.—a new waterproofing agent. It is said to have the special advantages that material treated with it has a soft feel and not the harshness commonly shown by waterproof fabrics. It is claimed that it mixes readily with cold water and that the impregnation liquors are very stable and do not show any foaming during the process of impregnation. It may be applied to animal, vegetable and artificial fibers. Circular I.G. 1159.

Chrome Fast Yellow 8 G L—a new brand which when dyed according to the chrome topped method produces bright greenish yellows, is said to be of excellent fastness to washing, fulling, carbonizing, decatizing and perspiration. It is said to be of very good solubility, to be extremely level dyeing and therefore very suitable for combination shades. The new brand is said to be of special interest for the production of bright green shades with chrome colors. Circular I.G. 1131.

Indanthren Brilliant Scarlet F R Supra fix—representing at present the brightest shade of scarlet available in the fast-to-light Indanthren range. This brand is said to possess excellent fixation and to be well suited for direct printing and for the production of bright scarlet discharges or for resist under Algosol dyeings or Aniline Black. Circular I.G. 1329.

Indanthren Orange RR Powder Fine for Dyeing—a new homogeneous dyestuff of the Indanthren class which will be of interest for the production of reddish shades of orange said to possess excellent fastness to light. It is said to be of particular interest for the dyeing of cotton-piece goods and on account of its ease of dispersion is highly recommended by the manufacturers for use in the pigment padding method. It is further recommended for the dyeing of rayon and pure silk. Circular I.G. 1351.

• SOAP CONSULTANT

Daniel Finkel, formerly connected with the chemical laboratory and technical sales force of the New Jersey division of the Armour Soap Works, has joined the staff of the Olive Oil Soap Co. and the Wollen Chemical & Supply Co., Paterson, N. J., in the capacity of consultant and special field representative on soap and allied products as associated with the textile industry in the metropolitan territory.

• HERCULES DIVIDEND

The Board of Directors of Hercules Powder Company on September 29th declared a regularly quarterly dividend of 1½% on its preferred stock. The dividend is payable on November 15 to stockholders of record November 4.

• CHINA'S DYESTUFFS TRADE

Military operations in and around Shanghai, China, have completely demoralized China's trade in dyestuffs, which centers about that port, according to cable advices from Commercial Attache Julean Arnold, Shanghai, made public by the Commerce Department's Chemical Division.

No dyes are entering or leaving Shanghai at present but cotton mills in the vicinity are believed to be adequately supplied as companies normally carry six months' stocks on hand, the cable stated.

Small quantities of dyes are reported to be moving into the interior through Canton in the south and Tsingtao and Tientsin in the north as conditions permit. Currency and trade conditions throughout central China are more nearly normal than elsewhere and dealers and importers are stated to be experiencing less difficulty in making collections in those regions, according to the American Commercial Attache.

In recent years China's cotton mills have made greater use of synthetic aniline and coal-tar fast dyes as the purchasing power of the masses increased. If present conditions obtain for any extended period, however, it is believed that the Chinese will fall back upon their old village economy and resort to greater use of natural vegetable indigo which can be produced in Szechwan and Kiangsi Provinces and in other parts of the interior, it was stated.

With the development of its large textile industry China became the most important dyestuffs import market in the world and the lagest single foreign purchaser of American synthetic dyes. Last year American exporters shipped 6,000,000 pounds of dyestuffs valued at $1,540,000 to China and during 1935 shipment aggregated 8,670,000 pounds valued at $1,935,000, statistics reveal.

In the first seven months of the current year American dye exports to China were recorded at 5,941,500 pounds, valued at $1,462,550, according to preliminary statistics.

• ANNUAL MEETING, USITR

The eighth annual meeting of U. S. Institute for Textile Research, Inc., will be held at the Hotel Commodore, New York, N. Y., Thursday, Nov. 4th, and it is hoped that this date will be reserved pending receipt of agenda and program, and that the latter will ensure attendance. In addition to the regular morning meeting of members for presentation of annual reports and election of directors there will be an open research conference in the afternoon, and a dinner in the evening, the latter open to members and their guests, including ladies.

It is hoped that at the Research Conference representatives of other bodies engaged in important textile research than U. S. Institute may be present and report on progress and future plans. There will also be an opportunity for members to suggest new research subjects to U. S. Institute's Research Council. For the dinner speaking program, and in addition to the opening address of President Garvan, the progress and results of two important textile researches are already promised: (1) A report by Dean Joseph H. Willitts and associates on Progress of the Survey of Production and Distribution Organization in the Textile Industries; (2) "Cellulose" Analyses, by Dr. Wanda K. Farr, director of the Chemical Foundation's research on the chemistry of cellulose at Boyce Thompson Institute for Plant Research.

U. S. Institute Research Progress

Three researches are now in progress: (1) The economic study now approaching completion at the Wharton School of Finance and Commerce, Philadelphia. This was suggested and organized by U. S.

Institute's Committee on Economic Research, and the Committee has acted in an advisory capacity to Dean Willits and his associates. The study is wholly financed by The Textile Foundation at $20,000. The report will be published in book form, and should be an important addition to the limited literature of textile merchandising.

(2) The research on warp sizing and sizing materials that has been in progress at Massachusetts Institute of Technology since May, 1935. Over $15,000 has been raised for this study, half having been granted by The Textile Foundation, and the balance furnished by co-operating "contributing" members of U. S. Institute. Six progress reports have been made available to co-operators, and a seventh report is nearly ready for mailing. These and additional reports are confidential to them, and to "contributing" members for this research until a year after completion of a study. This restriction as to publication of research results applies to all co-operatively financed researches, and has been found necessary to provide the needed financing. Eventually results will be made available to all members.

(3) The research on textile drying which has been in progress at the National Bureau of Standards since July 15, and is wholly financed at $5,000 by "contributing" members of U. S. Institute. The first confidential progress report of this study has already been received by co-operators.

All "contributing" members of U. S. Institute paying annual dues of $100 qualify to receive the confidential progress reports of one of the two co-operatively financed studies, and may receive those of the other co-operatively financed research by taking out another "contributing" membership. At present there are 15 firms having two "contributing" memberships for this purpose.

While U. S. Institute has no research foundation fund of its own at present, it may be of interest to note that the $30,000 available for its research this year is equivalent to the income on a 3% basis of a fund of $1,000,000.

Memberships

U. S. Institute's membership has shown a marked growth within the year, particularly that of the "contributing" class, the latter undoubtedly being due to increased interest in the co-operatively financed researches. Total membership of 308 is the largest paid membership of its career, as is its "contributing" membership of 112, the latter comparing with 69 last year, and the total with 265 in 1936.

Additional Services

Possibly a more accurate reflection of the domestic textile industry's interest in research is shown by circulation figures of U. S. Institute's monthly magazine, Textile Research. The latter totals 792, and with foreign subscriptions and the exchange list deleted aggregates 672. Of the latter 87 are paid domestic subscriptions, and 585 are member subscriptions, the latter number exceeding actual membership because each "contributing" member has the privilege of having any five of his representatives receive the magazine.

There is one special service rendered to members by the magazine that has shown marked expansion during the year, and that is the privilege of receiving photostat or other complete copies of articles abstracted. The character and limitations of this service are explained on the first page of the Abstract Section of each issue. Efforts are being made constantly to improve this service. Approximately 1,000 abstracts were published in the current volume, and during the last few months more than 100 complete copies of articles abstracted were inquired for.

CLASSIFIED

The rate for "Position Wanted" advertisements in this column is 2 cents a word—with a minimum of 50 cents per insertion. For all other types of advertisements—i.e., help wanted, machinery or supplies for sale—the rate is $5.00 per column inch or less per insertion.

WANTED: Manufacturer of a complete and highly developed line of finishing specialties for full fashioned hosiery mills is seeking the services of a representative with proven sales ability for North Carolina and Tennessee. Must have thorough knowledge of all hosiery dyeing and finishing problems with wide acquaintance and following in that territory. Salary, expense and bonus. Write fully giving age, qualifications, experience and references. All replies held in strict confidence. Write Box No. 980, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

WANTED: Wanted by a well-known manufacturer of sulfonated oils and other textile specialties chemist capable of doing research and development work. An excellent opportunity for a man with ideas and experience along this line. Write Box No. 977, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

WANTED: Skein Dryer—Conveyor Type. Must be in good condition. Write Box No. 982, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

POSITION WANTED: Chemist—graduate Lowell Textile, experienced, young, seeks position. Prefers field as research or control chemist in sulfonated oils or textile chemicals. Salary secondary. Knowledge German. Write Box No. 983, American Dyestuff Reporter, 440 Fourth Ave., New York, New York.

POSITION WANTED: By Textile Chemist experienced in the development and manufacturing of Sulfonated Oils, Softeners, Penetrants, Delustrants, Sizes, Splashproofing, Finishes, etc. Thoroughly capable of conducting or directing research and following products from laboratory scale to plant operation. Experience with Colloidal Mills and Homogenizers. Salary reasonable. Write Box No. 984, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.

REPRESENTATIVE WANTED: A well known organization of manufacturing chemists located within 100 miles of the metropolitan area is interested in getting in touch with a dyestuff concern who can represent their line of textile chemical specialties in the Middle West. All replies will be treated in strict confidence. Write Box No. 985, American Dyestuff Reporter, 440 Fourth Ave., New York, N. Y.